MICROWAVE BENEFICIATION OF COAL
TO IMPROVE GRINDABILITY
AND HANDLEABILITY

by

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ABSTRACT

Experimental results and analyses have shown that significant improvements in coal grindability (reductions in Relative Work Index) can be achieved by exposing coals to microwave radiation. Experimental data have indicated that low rank coals are highly responsive to microwave radiation, possibly due to their higher inherent moisture content. There is evidence to suggest that gaseous evolution (water vapour and volatile matter) and localised zones of differential expansion (arising for example from occluded mineral matter) in coal during heating give rise to crack formation and hence are the probable causes for the measured increase in coal grindability. The composition of the various coals treated by microwave radiation remained relatively unaltered and there was no significant change in coal calorific value or the proximate and ultimate analyses (dry, mineral matter free basis). Initial (laboratory-scale) microwave trials and pilot-scale test-work demonstrated an improvement in the grindability of various coals. However, the gross energy input for these tests were excessively high (220kWh/t) in comparison to that used mechanically for pulverised coal production (15-20kWh/t).

Improvements in microwave cavity design and increased electric field strengths may increase the energy efficiency of the process; however, further work would be required. Additional studies were carried out to evaluate the potential use of microwave technology for coal desulphurisation. The results were encouraging and show that substantial improvements in pyrite separation can be achieved with some coals.

Fundamental studies have shown that there is significant change in coal flowability following microwave exposure.

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CHAPTER ONE

MICROWAVE BENEFICIATION OF COAL TO IMPROVE GRINDABILITY AND HANDLEABILITY

1.1 Introduction-Microwave beneficiation of coal to improve grindability and handleability

The principle objective of this study was to investigate the effect of microwave radiation on the grindability and subsequent changes to the physico-chemical properties of a range of UK coals. Additionally, a subsequent aim was to ascertain any other beneficial effects (physical or chemical) of the microwave pretreatment of coal. In particular some confirmatory evidence was obtained concerning the use of microwave energy as a method of coal desulphurisation prior to combustion.

Previous studies have shown some improvements in coal grindability following pre-heat treatment (Harrison 1997). Changes in structure were attributed to the volumetric expansion of constituent mineral matter within coal promoting fracture propagation. Microwave energy can be applied as a source of heat treatment and several mechanisms can be proposed which may account for the improvements in coal grindability after microwave exposure. The organic components of coal are relatively poor absorbers of microwave energy. However, water is a good absorber of microwave energy and coals naturally contain water to varying degrees depending on their microstructure, geological location and relative humidity.

Water molecules are polar and when exposed to an alternating electric field; the molecules position themselves in the direction and at the same frequency to that of the applied field (Hulls, 1992). When a coal is exposed to microwave energy; the water molecules will undergo heating, change phase and expand creating internal pressures within the coal matrix, possibly weakening the structure. Similar effects would be expected from the expansion of some mineral constituents. However, each mineral has a specific heating rate when exposed to microwave radiation. Hence, the energy is not used to heat the whole of the material (in contrast to that of convective heating) but only those constituents which are considered good absorbers. Accordingly, this may improve the thermal efficiency.

Following the introduction of the principles of dielectric science and topics pertinent to microwave generation (Chapter 2), the thesis objectives were described, with particular reference to coal chemistry, applications, the generation and use of pulverised fuel and associated environmental regulations. A review of the UK coal production and consumption is presented in Chapter 3. Previous studies concerning comminution theory, thermally-assisted liberation and microwave-assisted comminution are reported in Chapter 4. Further cited literature is reviewed concerning the dielectric properties of coal, microwave pyrolysis, microwave assisted desulphurisation and the effect of microwave treatment on coal grindability (Chapter 5).

Chapter 6 outlines the fundamental experimental procedure for milling trials and microwave treatment, whilst Chapter 7 reports on the effect of coal rank on microwave treated coal grindability. Dielectric measurements are detailed and used to determine the penetration depth and heating rates of coals and associated minerals (Chapter 8). A combination of results (dielectric properties and data from laboratory milling tests) and fracture mechanics theory is use to hypothesize the possible mechanisms of structural weakening by microwave heating in Chapter 9. Pilot-scale test results and an economic evaluation of the microwave process are discussed in Chapter 10. The role of pyrite desulphurisation is introduced in Chapter 11 in terms of mineralogical analysis and magnetic separation test work; additional effects on coal following microwave treatment (pyrolysis and flowability) are also presented. Overall conclusions are given in the final chapter.

CHAPTER TWO

DIELECTRIC HEATING

2.1 Introduction-Dielectric heating

Microwaves are electromagnetic waves of a particular wavelength and frequency range situated between infrared and radio frequencies within the electromagnetic spectrum (Figure 2.1). Microwave technology to-date has been most often associated with domestic food preparation. The original uses in 1921 of high power microwave energy were for military purposes. In later years, microwave energy has been applied as an alternative to conventional convective and conductive heating and is used in many industrial processes. Conventional heating relies upon conductive heat transfer from the material surface to the interior; often associated with problems of cool centres and hot surfaces, especially with insulating materials. Microwave heating differs by means of the in-situ method of heating providing the prospect of uniform temperature distribution and improved heating efficiencies.

Materials differ in their ability to absorb microwave energy in an applied electric field. Each material has a particular optimum frequency which can be measured. However, in practice, for interference reasons the nearest practicable permitted frequency is used. High frequencies are used for many purposes: broadcasting, communications, navigation, and specific bands are allocated to industrial, scientific and medical purposes (Table 2.1).

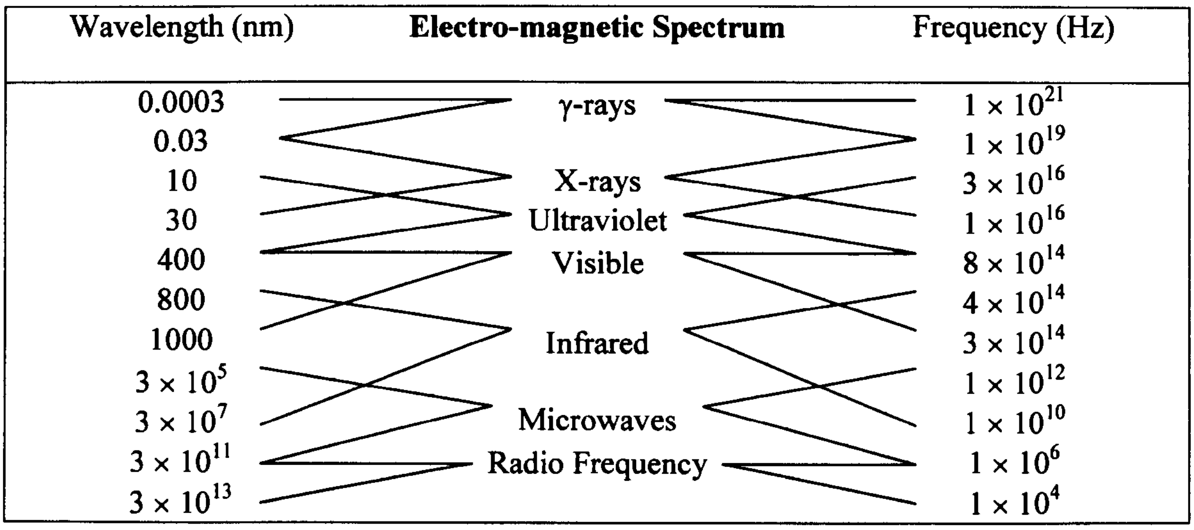


Figure 2.1 The Electro-magnetic Spectrum

Table 2.1 Frequency ranges allocated for industrial, scientific and medical purposes (Metaxas 1990)

Frequency (MHz)	Frequency tolerance ±	Area permitted
433.92	0.2%	Austria, Netherlands, Portugal, Switzerland, Germany
896	10 MHz	UK
915	13 MHz	North and South America
2375	50 MHz	Albania, Bulgaria, Czechoslovakia, Hungary, Romania, CIS
2450	50 MHz	Worldwide except where 2375MHz is used
3390	0.6%	Netherlands
5800	75 MHz	Worldwide
6780	0.6%	Netherlands
24150	125 MHz	Worldwide
40680	-	UK

2.2 Electric field

An electric field or voltage gradient can be simple to generate (connection of two parallel metal plates to a voltage supply, Figure 2.2). Using a direct current supply, the electric field strength is related to the applied voltage and distance between the positive and negative plates (Equation 2.1).

$$E=V/D \tag{2.1}$$

E	Electric field strength (v/m)
V	Voltage (v)
D	Distance (m)

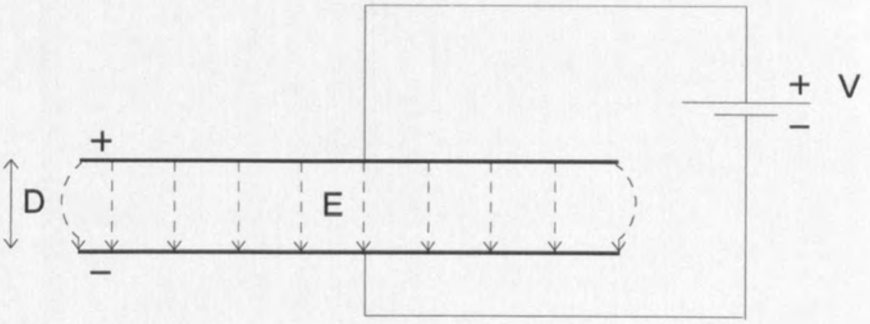


Figure 2.2 Electric field generator-parallel plates (Von Hippel 1954)

An AC voltage can produce an electric field which alternates at the same frequency as the source. Microwave fields are generated when the frequency of the source (or the field) is between 300 MHz and 300 Hz producing corresponding wavelengths of 1m and 1mm respectively (Harrison 1997).

2.3 Polarisation

When a material is placed within an alternating electric field (radio – microwave frequencies) the material will polarise depending upon its molecular structure. Polarisation follows the direction of the electric field and realigns at the same frequency. This realignment of the molecules or shift of electron matrix produces a frictional in-situ mode of heating. There are four principal forms of polarisation (dependent on material properties and the applied electric field) that are affected the most by microwave fields and are believed to be responsible for microwave heating.

Electron polarisation arises when the material has a positively-charged centre or nuclei surrounded by a negatively-charged electron cloud. When the material is placed within an electric field the electrons are displaced producing a temporary dipole movement (metallic bonded molecules).

Atomic (or Ionic) polarisation occurs due to the distortion of atoms within molecules of ionic molecular bonds. The applied electromagnetic field acts upon these molecules causing their charge equilibrium position to change, inducing polarisation.

Orientation polarisation is the realignment, by an applied electromagnetic field, of molecules already permanently polarised (dipole molecules) due to their chemical bonds (water molecules). This form of polarisation is most significant at microwave frequencies (Figure 2.3).

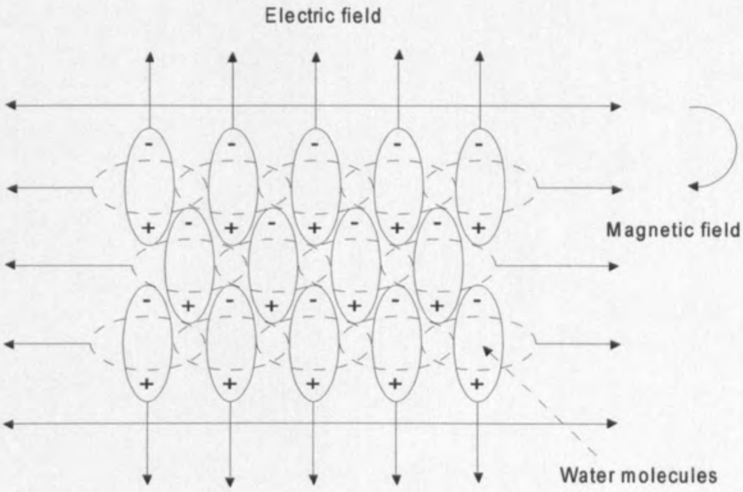


Figure 2.3 Water molecules exposed to microwave radiation (Rowson 1986)

Space charge polarisation is the accumulation of charge and migration of charge under the influence of an applied electromagnetic field. Whilst the effect is significant at radio frequencies, it may also occur at microwave frequencies (Hulls 1992).

The sum of these forms of polarisation (Atomic polarisation α_a, Electronic polarisation α_e, Orientation polarisation α_o, and Space charge polarisation α_s) is defined as the resultant polarisation ability (α) of the dielectric material i.e.

$$\alpha = \alpha_a+\alpha_e+\alpha_o+\alpha_s \qquad (2.2)$$

2.4 Electric permittivity and dielectric constant

The ability of a dielectric to be polarised is expressed in terms of the 'electric permittivity' of the material (Equation 2.3). The real permittivity is defined as the ratio of the capacitance C of a cavity filled with the dielectric material to the capacitance (C_o) of the same cavity under vacuum (Chelkowski A 1980) i.e.

$$\varepsilon' = \frac{C\varepsilon_o}{C_o} \qquad (2.3)$$

ε'	Real permittivity (F/m)
ε_0	Permittivity of free space (F/m)

This would only occur if the realignment of molecules is at the same frequency and in phase with the applied electric field. Most materials exhibit a difference in phase, in which the polarisation movement lags behind the alternating electric field frequency and indicates a loss in conductivity within the material that fills the cavity (relation to currents within the material out of phase with the electric field). To account for this change in conductivity, the electric permittivity is described as a complex quantity (Equation 2.4) with both real and imaginary parts (the imaginary part accounts for the loss in conductivity in the material):

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad (2.4)$$

ε''	Imaginary permittivity (F/m)
ε^*	Complex permittivity (F/m)
j	$\sqrt{-1}$ (imaginary complex number)

The relationship between the real and imaginary parts is expressed as the loss tangent, this refers to the angle (loss angle) the material is out of phase with the electric field. The sum of the real and imaginary parts represents the complex permittivity and the loss angle is that between the electric field and the complex permittivity. The real permittivity is considered to be in phase with the alternating electric current and the imaginary permittivity represents the phase lag i.e. the tangent of the angle between the sum of the two parts (complex permittivity) and the real permittivity is equal to the imaginary part divided by the real. The loss tangent (tan *d*) can be used to represent the fraction of stored energy lost per period of field oscillation:

$$\varepsilon'' = \varepsilon'.\tan d \qquad (2.5)$$

The parameters can be evaluated, the real permittivity can be measured from the capacitance ratio described in Equation 2.3, and the imaginary permittivity is evaluated from measurements of the loss angle. However, their magnitudes are generally low (approximately 10^{-10} F/m) and the components are usually re-scaled by dividing the permittivities by that of free space (8.85×10^{-12} F/m). The resulting values are termed the relative real permittivity (or relative dielectric constant) and the relative imaginary permittivity (or relative loss factor).

2.4.1 Loss factor and dielectric conductivity

The ability or the receptiveness of a material to heating is known as the 'loss factor', the higher the value of the loss factor the more receptive the material is to electric fields. In general, materials with relative loss factors greater than 0.02 are considered possibly candidates for dielectric heating. The ability or receptiveness to microwave heating is often expressed in terms of the dielectric conductivity (Equation 2.6), which can be calculated from known values of the loss factor and the applied frequency.

$$\varepsilon'' = \frac{O_c}{\omega} \tag{2.6}$$

O_c	Dielectric conductivity (s/Ω)
ω	Angular frequency (s^{-1})

2.4.2 Power absorption

The power absorbed per unit volume by a given material in a microwave field is related to the electric field strength, dielectric conductivity and corresponding magnetic field strength (Equation 2.7). The magnetic field strength is usually considered negligible unless the material has a high magnetic susceptibility.

$$P = 2.\pi.f.\varepsilon_r''.\varepsilon_0.\mathrm{E}^2 + 2.\pi.f.\mu_0.\mu''.\mathrm{H}^2 \tag{2.7}$$

P	Power absorbed (W/m^3)
f	Frequency (Hz)
ε_r''	Relative loss factor or relative imaginary permittivity
ε_0	Permittivity of free space (F/m)
H	Magnetic field strength (N/A^2)
μ_0	Permeability of a vacuum (N/A^2)
μ''	Permeability (N/A^2)

Substituting into (Equation 2.7) with (Equation 2.6) and assuming that the magnetic susceptibility effect is negligible, the power per unit volume can be expressed as:

$$P = 2.\pi.f.\mathrm{E}^2.\varepsilon_0.\varepsilon_r'' \tag{2.8}$$

It is can be seen from Equation 2.8 that the power absorbed is proportional to the square of the electric field strength i.e. $P \propto E^2$. This indicates that the generation of high electric field strengths represents a possible method of rapidly accelerating power absorption.

2.4.3 Heating rate

The theoretical heating rate of a specific material within a microwave cavity can be evaluated providing the dielectric and physical properties of the material and microwave are known. Using accepted standard theories regarding heating rates i.e.

$$\mathrm{Q} = M.C_p \frac{\theta}{t} \qquad (2.9)$$

Q	Energy absorbed or produced (W)
M	Mass of the body heating (kg)
C_p	Specific heat capacity (J/kg)
θ	Change in temperature (°C)
t	Change in time (s)

Manipulating this (Equation 2.9) and substituting (Equation 2.8) for the power absorbed a theoretical heating rate can be expressed in terms of dielectric properties.

$$\frac{\theta}{t} = \frac{\omega.\varepsilon_r''.\mathrm{E}^2.\tan d.\varepsilon_0.V}{M.C_p} = \frac{\varpi.\mathrm{E}^2.\ \varepsilon''.}{\rho.C_p} \qquad (2.10)$$

ρ	Density of material (kg/m^3)
V	Volume of material (m^3)

2.4.4 Penetration depth

Using dielectric principles, relationships for penetration depth of microwave radiation have been proposed (Metaxas 1983).

$$D_p = \frac{\lambda.\varepsilon_r^{'1/2}}{2\pi.\varepsilon_r^{''}.} \tag{2.11}$$

D_p	Penetration depth of wave (m)
ε_r	Relative dielectric constant or relative real permittivity
λ	Wavelength (m) $\lambda = c/f$
c	Speed of light (m/s) 3×10^8 m/s
ε_r''	Relative loss factor or relative imaginary permittivity

2.5 Factors effecting the dielectric field

A number of controlling factors affect a material's ability to absorb microwave radiation within a dielectric field. In particular these are, the material temperature, chemical composition and the frequency and strength of the applied electric field.

2.5.1 Variations of dielectric properties with changing temperature

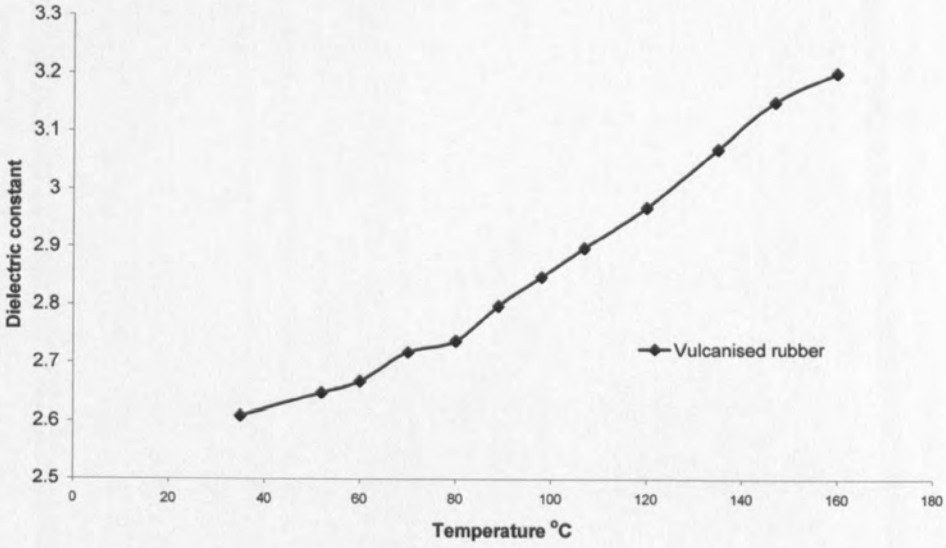


Figure 2.4 Dielectric properties of vulcanised rubber with temperature (Catala-Civera 1997)

An increase in temperature increases dipole mobility, where in additional energy is adsorbed and hence frictional heat produced. The dielectric constant also increases with temperature as shown in Figure 2.4. However, if the material continues to absorb energy after the 'critical temperature' (unique to a specific material) heating can lead to a 'thermal run away' system i.e. the heating rate increases exponentially until material deformation (Church et al 1988). Some materials have very low microwave heating rates, however, at elevated temperatures some materials (ceramics) couple with microwaves and absorb microwave energy. Such materials would require initial heating from another source until the particular temperature is reached for microwave susceptibility, after which the material is receptive to microwaves and can be further heated by microwave energy.

2.5.2 Effect of microwave frequency on dielectric properties

Dielectric constant varies with frequency and those for specific minerals may resonate at a particular frequency. The latter are considered as the optimum value for the material absorption capability. Resonant frequencies depend upon the dielectric properties of the material, and hence it's physico-chemical characteristics (composition, moisture content, mineral matter distribution, crystal structure, particle size, and density/voidage).

2.5.3 Chemical composition and distribution

Natural materials may contain a range of chemical compounds which may vary in their receptiveness to microwave radiation. Hence, both the chemical composition and distribution of receptive compounds will affect the overall heating of a material. The latter can be enhanced by 'doping' (blending) materials which show a poor propensity for microwave heating with those having more receptive features to improve heating.

2.5.3.1 Water content

Water is a good absorber of microwave radiation and the drying of wet materials can be highly efficient by microwave heating; more so than by convective heating and, in particular, if the material contains inherent moisture and where temperatures exceeding 100°C are required. Similar principles apply with regard to net heating effects (Section 2.5.3).

2.5.3.2 Particle size

Some authors report that loss factors increase linearly with decreased particle size (Andres 1988, Walkiewicz 1988); other workers claim their heating rates vary according to material and particle size range. Standish et al (1989) demonstrated the effect of particle size on heating rates on silica. Heating rates showed an initial decrease with increasing particle size (0.2-0.5mm) and a subsequently increase with increasing particle size, peaking at ~1.5mm, particle coarser than 1.5mm showed further reductions in heating rates.

2.6 Microwave technology

The majority of industrial microwave units consist of three components. The generator, which converts the power supplied into microwave energy, the waveguide which carries the energy to the applicator from which the radiation is distributed to the product.

2.6.1 Microwave generator (The Magnetron)

There are various forms of microwave generator including the magnetron, klystron and solid state devices. The magnetron generally represents the most suitable compromise between power output, efficiency and cost. The device has an oscillating diode which produces electrons that are accelerated by an electric field between positive and negative electrodes. A magnetic field deviates the trajectory of the electrons producing a high frequency oscillation. An antenna emits the electromagnetic field to the waveguide where it is then directed to the applicator. A water cooling system protects the magnetron from reflected energy (Hulls 1992). A typical electric circuit for microwave generation is shown in Figure 2.5.

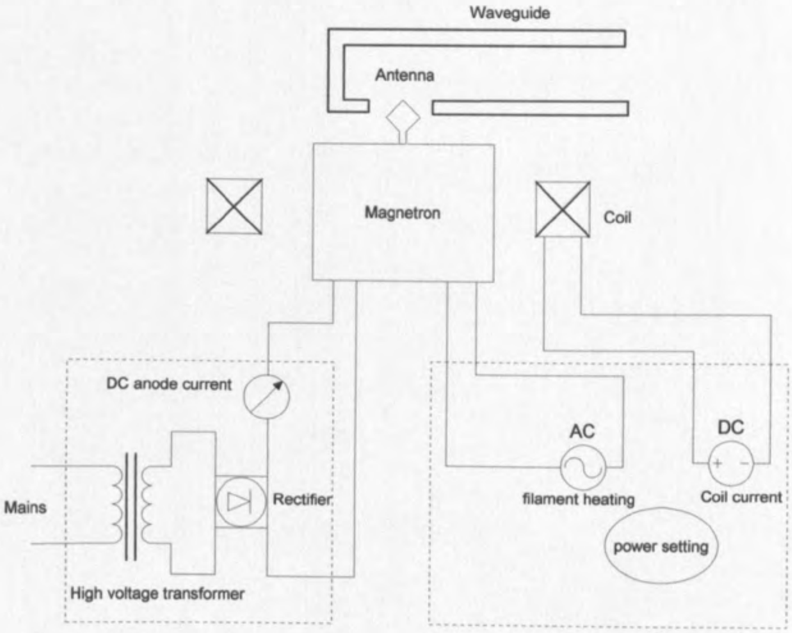


Figure 2.5 Magnetron electric circuit (Hulls 1992)

Magnetrons are usually designed to operate at standard frequencies; their power output and efficiency vary depending upon the frequency and design. In particular, magnetrons with an operating frequency of 2.45GHz have a typical power output between 0.6 to 6kW and an overall efficiency of 50%-60%. A greater efficiency (80%-90%) can be achieved with magnetrons of a frequency range 896/915MHz, these magnetrons can also provide higher power outputs (up to 60kW).

2.6.2 Waveguides

The purpose of waveguides is to direct the incident microwave to the load (material absorbing the microwave energy). Waveguides are generally in the form of rectangularly shaped truncking fabricated from conducting materials (copper, brass or aluminium); typically, metals reflect microwaves and absorb little energy.

2.6.3 Incident and reflected power, circulators

If the incident microwave is totally absorbed by the load (i.e. assuming negligible losses in the waveguide) then the load can be considered as 'matched'. However, if some of the incident wave is reflected from the load; then reflected radiation may travel back to the generator and give rise to a mismatched load whereby the incident and reflected waves interfere and produce standing waves. The latter lead to significant localised increases in electric field strength that may cause overheating of the generator or give rise to arcing near the antenna. As reflected microwaves can reduce the operating life of the generator, industrial applicators are equipped with circulators. The latter redirect the reflected wave to another absorber (another matched load, usually a water-cooled) to prevent reflected waves reaching the generator.

2.6.4 Applicators

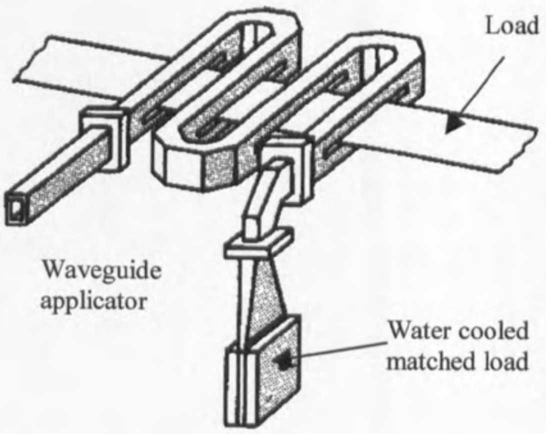


Figure 2.6 The progressive waveguide applicator (Hulls 1992)

An applicator is that part of the microwave equipment whereby the electromagnetic energy is transferred to the load. The applicators vary in design (depending on microwave use) and operating features (size of the load and whether the process is continuous or batch). To prevent leakage of radiation most applicators have the appearance are of the form of metallic boxes designed to reflect the electromagnetic energy within them. The progressive wave applicator (Figure 2.6), mono-mode cavity (Figure 2.7) and multi-mode cavity (Figure 2.8) are the most common design of applicators. The progressive wave applicator has a folded rectangular waveguide that the load continuously passes through absorbing the energy

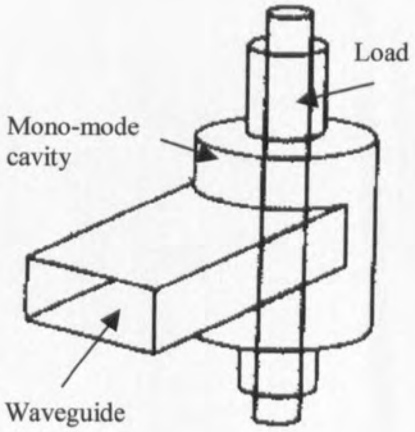


Figure 2.7 The mono-mode cavity (Hulls 1992)

Similar in design to the progressive waveguide applicator, the resonant or mono-mode applicator (Figure 2.7) also produces highly homogenous electric field distributions. The mono-mode applicator is generally used on poor dielectric heaters as they create localised increases in electric field strength within the load.

Domestic microwave cavities are examples of the multi-mode applicator. As shown in Figure 2.8, the electromagnetic energy is continuously and randomly reflected from the metallic walls of the applicator. The electric field thereby fills the whole cavity so that the load will absorb energy wherever it is located. However, due to interference between multiple reflections, localised increases in electric field strength may lead to uneven heating. As a means of compensation, turntables and mode stirrers are used to periodically change the field distribution within the cavity and produce more uniform heating.

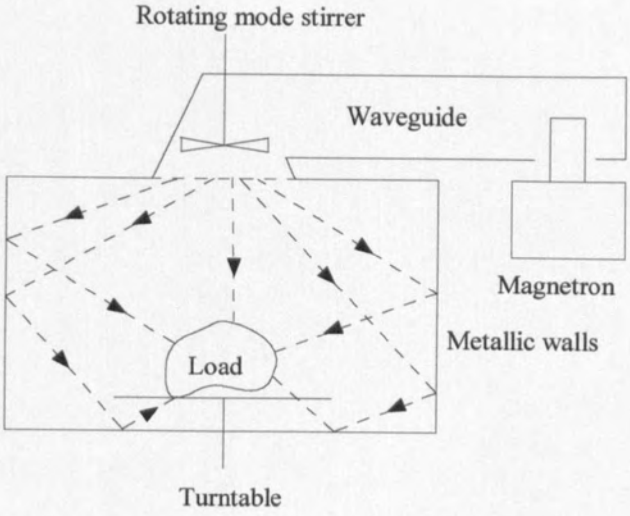


Figure 2.8 The domestic (multi-mode) microwave applicator (Hulls 1992)

CHAPTER THREE

COAL CHARACTERISTICS AND PHYSICAL PROPERTIES

3.1 Introduction-Coal characteristics and physical properties

This section describes those principle physico-chemical properties of coals considered to affect their structure, heating characteristics and breakage characteristics. A brief summary of the current UK coal market and prospects are discussed.

3.2 Coal structure

Coal is a complex, heterogeneous, hydrocarbon-based mineral which has been formed over many years by a succession of changing geological stages. The initial (diagenetic) stage is represented by an accumulation of active peat and this is followed by a metamorphic stage arising from changes in the geological features. In particular, overlaying and geothermal stresses effect the coal physical structure and its chemical properties (moisture and volatility). The metamorphic process progressively converts the original accumulated peat to a material of a similar structure to that of graphite.

Coals are ranked according to their extent of metamorphism. Typically, the older the coal the higher the rank, anthracite and lignite may be designated as high and low rank coals respectively. The most useful properties of the coal which define its rank are considered to be the carbon content, volatile matter and moisture content together with the calorific value (Figure 3.1). Coals are ranked into four main groups of increasing rank:

(Low rank) Lignite →Sub-bituminous→Bituminous→Anthracite (High rank)

	Low rank ——→ High rank
Rank	Lignite → Sub-bituminous → Bituminous → Anthracite
Age	——→ Increases ——→
Carbon content %	——→ Increases ——→
Volatile matter %	——→ Decreases ——→
Moisture content %	Decreases ——→ → Increases
Calorific value	——→ Increases ——→

Figure 3.1 Dependency of coal property on rank (Elliot 1981)

Each of the four main groups of coal rank (Figure 3.1) are split into sub-groups which are usually defined according to the physical and chemical properties of the coal. Figure 3.2 shows the variation in coal moisture content, volatile matter content and fixed carbon (ash-free basis) for a wide range of coals.

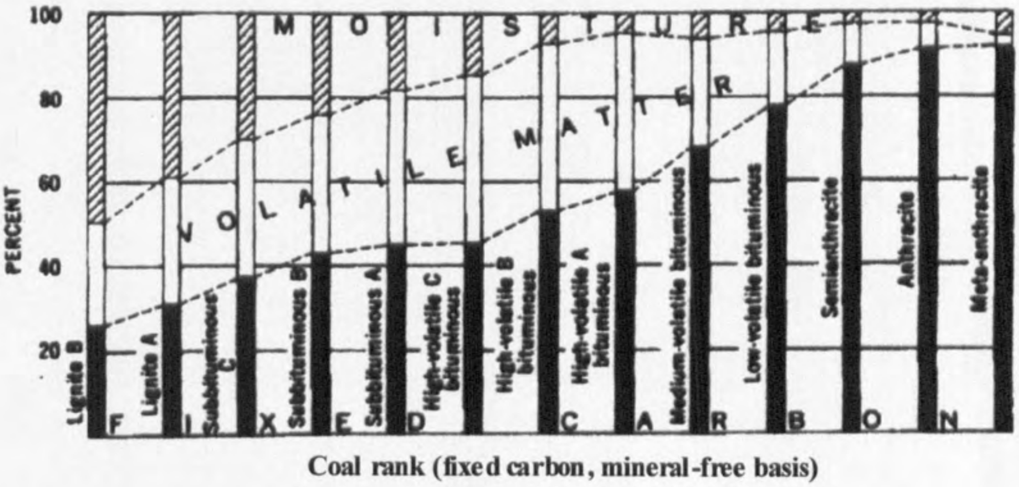


Figure 3.2 Coal rank based upon mineral matter free proximate analysis (Elliot 1981)

The ash content of coal although a major feature in some coal process utilisation is not considered as a measure of coal rank. Ash content is a measure of the oxidised products of the associated mineral matter constituents, these generally include eleven mineral compounds: pyrite, ankerite, gypsum, dolomite, apatite, calcite, mica, kaolin, quartz, rutile and siderite.

The calorific value of coal is defined as its energy release associated with complete combustion and represents an effective way of expressing the fuel potential of the coal.

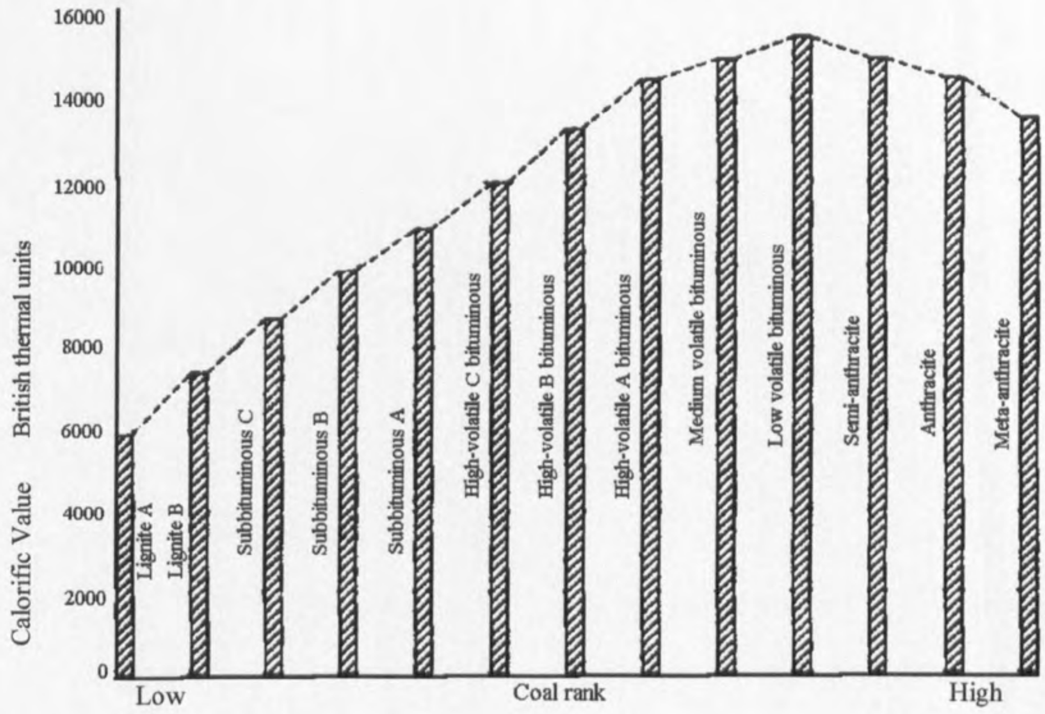


Figure 3.3 Calorific value against coal rank (mineral matter free basis) (Elliot 1981)

Calorific value generally increases with coal rank (Figure 3.3). Usually the main components controlling the energy release capabilities are moisture, volatile matter and total carbon content. Increasing moisture reduces the calorific value as seen for low rank coals; increasing volatile matter tends to increase the calorific value and this leads to the feature of medium rank coals having a slightly higher heating value than high rank coals.

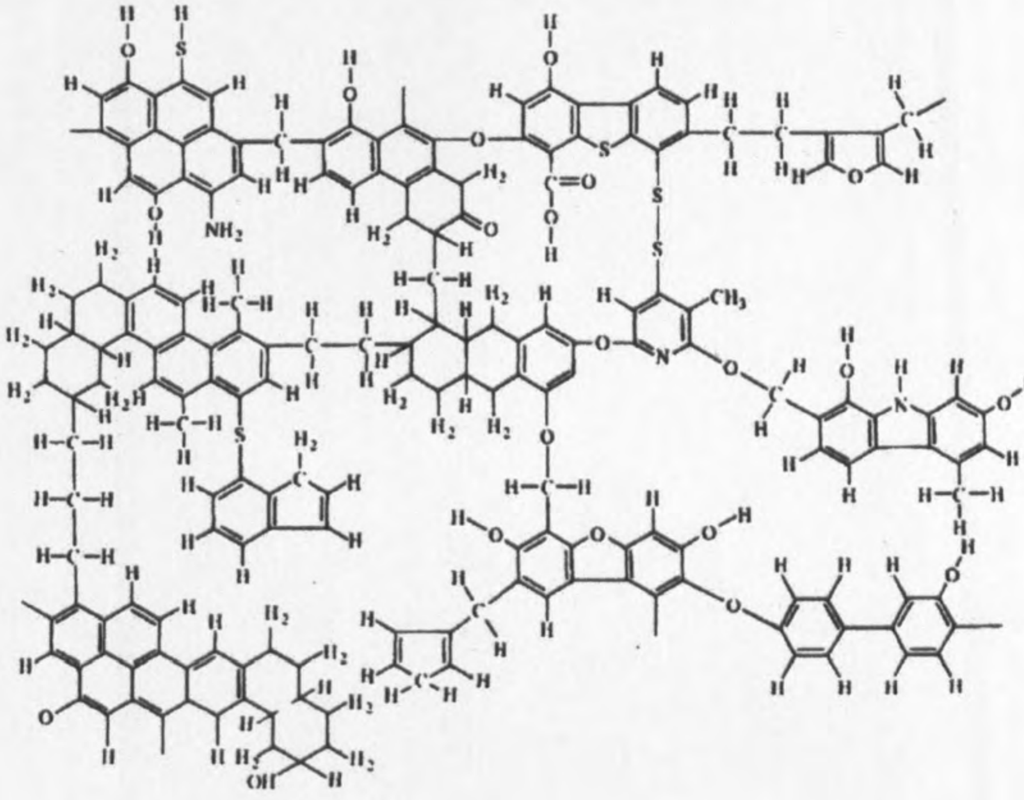


Figure 3.4 A proposed model of coal structure (Elliot 1981)

Coal is a heterogeneous material in a transitional phase between peat and graphite. Due to the diverse conditions under which coals are formed, they can be considered as individuals in structural terms; furthermore, coal from the same seam can vary significantly in composition and therefore structure. Whilst a model structure of organic coal can not truly represent all coals, models have, however, been proposed from chemical studies and x-ray analyses (Figure 3.4) which identify trends between the common bonds found within organic coal. Organic coal is composed mainly of aromatic and hydroaromatic groups linked by functional hydrocarbons. The functional hydrocarbon groups are believed to be the structures that break or join aromatic groups during coal pyrolysis and coalification. Studies have indicated that the number of linked aromatic groups increase with the rank of coal, the greatest transition being between bituminous and anthracite coals. This may be one of the reasons why the grindability increases with medium coal rank (Section 3.4).

3.3 Porosity, Surface area and Density

Coals are porous materials. Porosity and its dependence on rank are significant features in so far as porosity can affect coal specific gravity, surface area and the ability to absorb or diffuse gases into or from the material. Pore diameter can vary from less than 20Å (for micropores) (1Å = 1000µm) to about 200Å or more for the larger channels or capillaries (macropores). Gases of differing molecular weight are used to determine the surface-areas associated with the pore classification (i.e. nitrogen at –196°C is used to measure the macropore volume and helium, carbon dioxide, methanol, neon or xenon at low temperatures are employed to determine the total open pore volumes). Studies on American coals have shown that the porosity of coals of fixed carbon contents less than 75% (dry, mineral matter free basis) are predominantly due to macropores. The pore structure of coals with fixed carbon

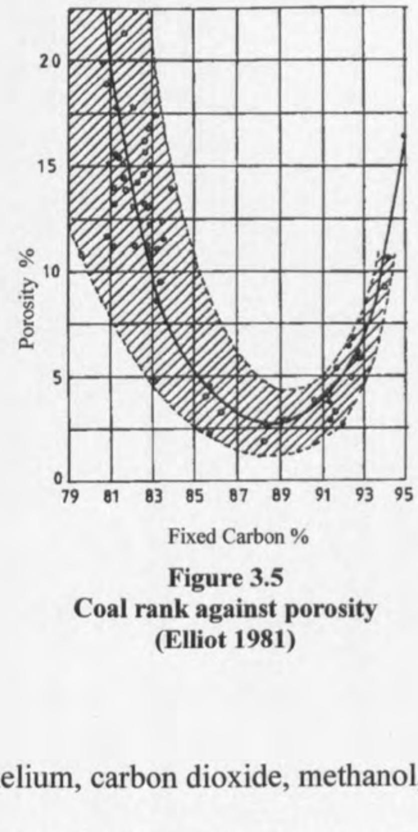


Figure 3.5
Coal rank against porosity
(Elliot 1981)

contents between 75% and 84% are predominantly of micropore and transitional pore sizes; for coals of a fixed carbon content greater than 85%, the pores are predominantly of micropore sizes (Elliot 1981). Whilst the total surface areas measured by the use of different gases are often different, similar trends with coal rank are evident and the surface area is primarily related to coal porosity. Low rank coals have a high porosity and high surface area, which decrease as coal rank increases to approximately 85-90% fixed carbon content; subsequently, porosity and surface area increases with increasing rank (Figure 3.5).

3.4 Hardgrove Grindability Index

Several methods of determining coal grindability have been proposed, all based on the use of roll crushers or ball mills. The most frequently used methods are those of Bond (1961) and Hardgrove (1932). The latter test has become preferred (in view of mechanical reliability of the mill and the ease of data evaluation) and in, 1951, was adopted in a standardised form by ASTM Standards Committee (ASTM D409). In use, a sample of coal of specified size range is charged into a captive ring-loaded mill which is subsequently operated at a constant speed for a given time. The ensuing increase in coal specific surface area is measured and compared with that of a standard coal (Pennsylvania Upper Kittaning seam) which has an assigned Hardgrove Grindability Index (HGI) of 100. A coal that can be broken extensively has a high HGI.

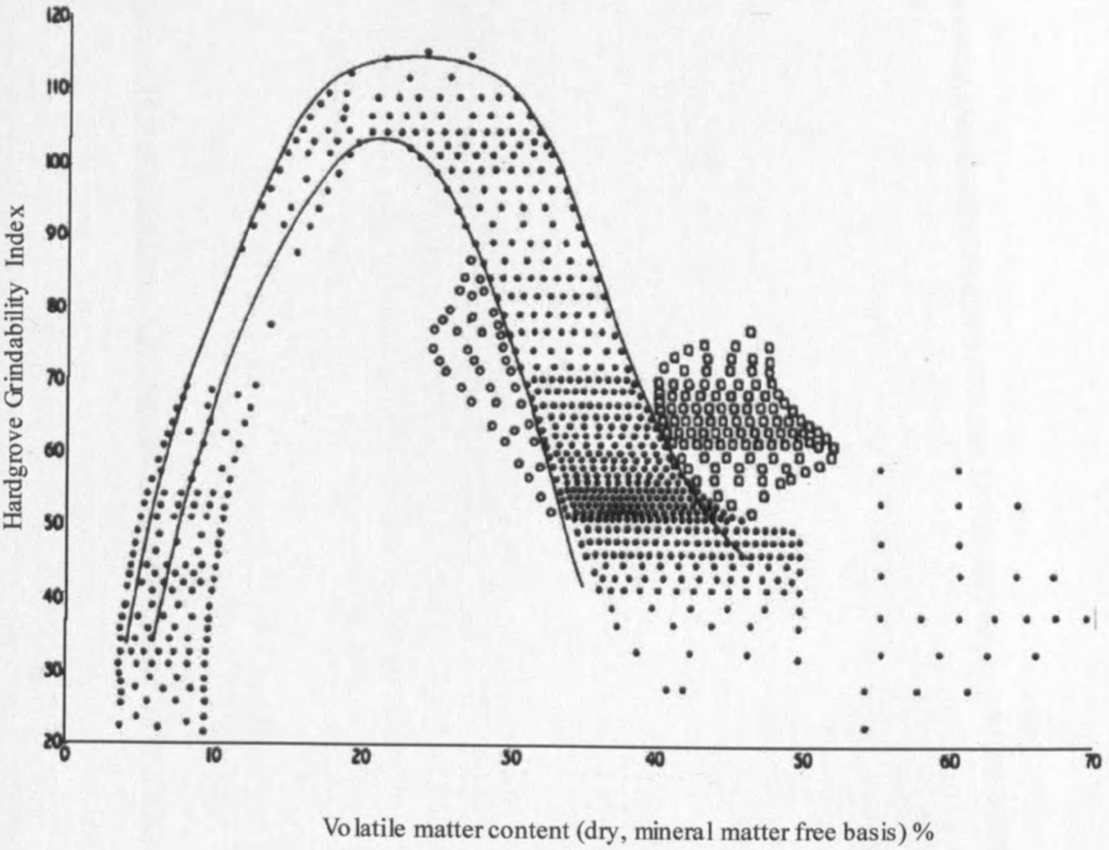


Figure 3.6 Hardgrove Grindability Index against rank (Elliot 1981)

The variation of Hardgrove Grindability Index with coal rank (Figure 3.6) indicates some scatter, particularly for low rank coals. Possibly, the structure of 'younger' coals (Lignite) may be such that they are difficult to grind by conventional milling methods due to their moisture content. For fully-formed coals (with the volatile matter between 7-25% dry, ash free basis) the structure becomes more compact as rank increases and accordingly, energy requirements for milling become higher.

3.5 Flowability

This section describes those properties of coal which have a major effect on its bulk flow characteristics. Particle size and shape are significant, and more generally, assemblies of large, smooth or spherical particles flow more readily than fine, angular particles (which can interlock or agglomerate in the presence of moisture or clay). In particular, free moisture tends to promote agglomeration of fine particles, making the coal cohesive. Usually, the most adversely affected coals are those of low rank or with moisture contents between 20-30%. Furthermore, soft (high HGI) or friable coals can break down into fines which could collect moisture. Oxidised or weathered coals generally have higher moisture retention abilities and finer particle sizes, which again lead to poor flow characteristics. Mineral components may also adversely affect coal flow properties and, in this respect, clays are significant, particularly the swelling type e.g. bentonite. Additions of emulsions, dust suppressants and flotation reagents can also have an adverse affect on the flow characteristics of coals and, in turn, these properties relate to the bulk handling and storage characteristics of coals. In particular increased coal storage time and consolidation pressure in bunkers and surface characteristics of coals (oxidation etc) can promote flow problems.

3.6 Coal dependent utilities

Coal remains the principle source of foundry and metallurgical coke used in the production of iron and steel. Other industries such as cement, brick, fireclay, refractories, glass and other chemical industries are also consumers. However, the coal consumption in these industries is far less than that of power generation. Additionally, domestic coal combustion has declined in Europe and North America since the introduction of clean air legislation and the use of other fuels for space heating (gas, oil and electricity).

Within the United Kingdom, the current major market for coal is power generation in spite of competition from natural gas and oil.

3.7 The current UK coal situation

UK coal production for industrial and domestic coal combustion has declined in response to demand. In some cases, electricity has been used directly and in others, alternative, cleaner fuels have been used in compliance with government legislation. Power generation (pulverised fuel firing-PF) remains the major consumer of UK coal but tonnage have been reduced (significantly since 1995) because of competition from other fuels.

New technologies are required to maintain competitiveness. Major outstanding problems in fine coal combustion (particularly gas emission levels) are receiving much attention with regard to the use of cleaner coal and/or improved combustion control technologies. Furthermore, reductions in process energy requirements would also benefit existing power stations. It should be appreciated that many current PF power stations have considerable operational life expectancies and new technologies would be most suitable if they can be integrated into existing plant.

3.7.1 UK coal production

Coal production has declined since 1970 with a sharp fall following the privatisation of the National Coal Board in the early 1980's. Imported coal and cheaper mining methods (e.g. opencast) are now necessary to ensure that coal can compete with other commercial fuels. In particular, imported coals now provide up to 30% of the coal consumption in the UK and opencast mining has had sustainable production since 1980 (Table 3.1).

Table 3.1 UK coal production (million tonnes) (D.T.I 1999)

Mining method	1970	1980	1990	1996	1997	1998
Deep mined	136.7	112.4	72.9	32.2	30.3	25.0
Opencast	7.9	15.8	181	16.3	16.7	15.0
Total (including slurry)	**147.2**	**130.1**	**92.8**	**50.2**	**48.5**	**41.4**

3.7.2 Coal consumption in the UK

Cleaner and less costly energy sources have replaced coal in many of the former consuming sectors such as industrial, commercial and domestic combustion. During the last thirty years, consumption had fallen in these combined sectors from 79.7Mt (1970) to 14.6Mt (1998) (Table 3.2). The electricity industry is currently the main consumer of coal within the UK although, with the introduction of natural gas (cleaner, cheaper fuel), the use of both oil and coal has declined in recent years. Coal consumption within the power generation industry may have stabilised with the current economic situation for PF power generation, however it is not expected to increase unless new technologies improve efficiency.

Table 3.2 Coal consumption in the UK (million tonnes) (D.T.I 1999)

Sector	1970	1980	1990	1996	1997	1998
Power stations	77.2	89.6	84.0	54.9	47.2	48.3
Domestic	20.2	8.9	4.2	2.7	2.6	2.2
Industry	19.6	7.9	6.3	3.6	3.0	2.7
Services	4.2	1.8	1.2	0.6	0.6	0.3
Other energy industries	35.7	15.3	12.5	9.6	9.6	9.4
Total consumption	**156.9**	**123.5**	**108.3**	**71.4**	**63.1**	**62.9**

3.7.3 Electrical power generation, type of fuel

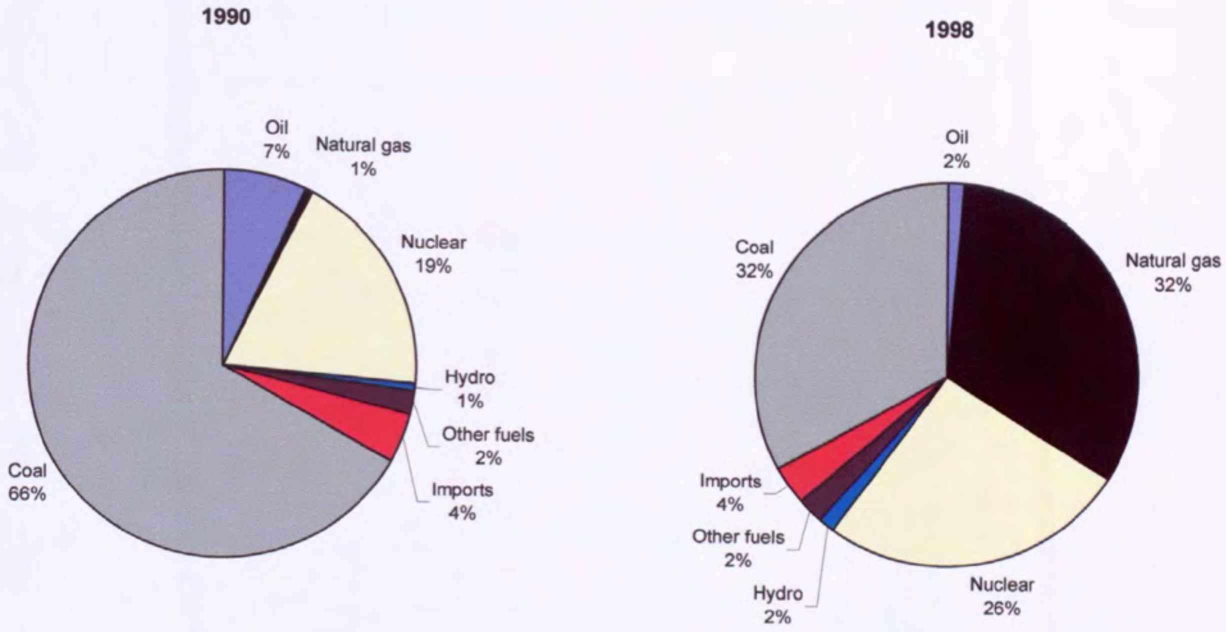


Figure 3.7 Electricity supplied by fuel type (D.T.I 1999)

Over the last decade, power generation in the UK has dramatically changed in preference to cleaner, cheaper and more thermal efficient fuels (Table 3.3). Oil use in power stations has decreased primarily due to its high cost and increased requirements in the automotive industries (crude oil production and petroleum consumption has increased 133Mt and 78.4Mt respectively) and exports. It is also noted, that proven and estimated offshore reserves of natural gas and oil at current consumption levels will last an estimated 50 years (D.T.I 1999). Increasing efficiency and advancing technologies have influenced the increase in nuclear power generation. The introduction of biofuels (landfill and sewage gases, domestic wood, industrial wood, straw and refuse combustion) and other renewable fuel sources have had an effect on the total energy production. More significant, however, is the increase in nuclear power. These changes have been associated with significant reductions in the use of coal (~50% since 1990) and oil (~75% since 1990) for power generation and consumption (D.T.I 1999).

Table 3.3 Electricity supplied by fuel type and consumption (TWh) (D.T.I 1999)

Fuel type	**1980**	**1990**	**1996**	**1997**	**1998**
Coal	190.0	207.9	139.6	114.7	117.1
Oil	33.9	21.1	12.9	7.5	5.3
Gas	1.6	1.6	80.9	106.1	114.0
Nuclear	32.3	58.7	85.8	89.3	91.2
Hydro	7.3	7.9	3.3	4.0	5.1
Other fuels (Biomass etc)	-	-	5.7	6.0	6.2
Imports	-	11.9	16.7	16.6	12.6
Total	**265.1**	**309.0**	**344.9**	**344.2**	**351.5**
Total consumption	**241.6**	**284.4**	**314.3**	**317.5**	**324.3**

3.8 Pulverised fuel power generation

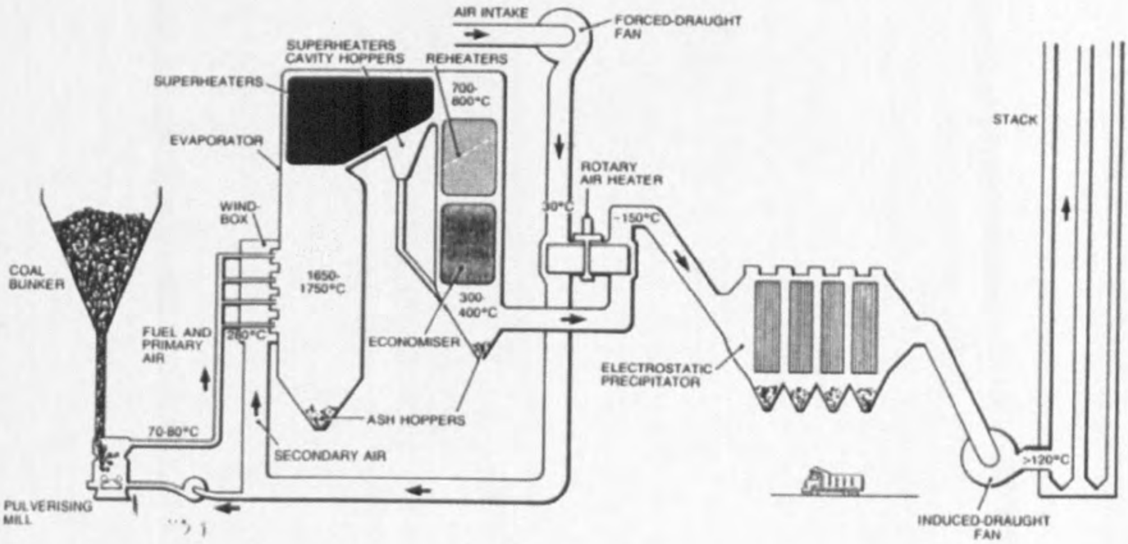


Figure 3.8 Coal fired boiler circuit (C.E.G.B. 1971)

The design features of power station boiler plants (Figure 3.8) depend, in part, on the type of pulverised coal used for firing. For anthracitic coals (of low reactivity and high ignition temperatures) the 'downshot' (long flame) combustion chamber is preferred in order to maximise the coal residence (burning) time. A further feature may be the provision of a refractory arch and delayed secondary air admission to maintain ignition stability. For bituminous and sub-bituminous coals, front-wall or corner-fired combustion chambers can be used. The required coal residence time is relatively short (~2 seconds) and swirl burners can be used (with mixing of the primary and secondary air close to the nozzle) to promote short flames.

With regards to the milling plant, primary air (at about 80°C) is ducted into the mill circuit to convey the ground coal to the burners. Part of this air can be used for classification purposes i.e. returning oversize coal to the mill for further grinding. Radiant heat generated by the flames in the combustion chamber is absorbed, in part, by the water walled (evaporative) surfaces of the combustion chamber and the balance being absorbed by the radiant superheater (steam) at the chamber outlet. Consequently, exit gas temperatures are reduced and the subsequent heat transfer to the air-preheater and economiser are predominantly by forced convection. The exit flue gases are maintained above 120°C to prevent corrosion i.e. SO_2 and SO_3 reacting with moisture producing sulphuric acid.

3.9 Size distributions for power generation

Typically, power station milling plants are designed for an upper limit of coal size not exceeding 40-50mm. The majority of UK bituminous coals are milled to a fineness of 70% ≤ 75μm, sufficient to ensure complete combustion. The mill power required is about 15-20kWh/t and is usually within acceptable levels (Table 3.4). For the lower volatile characteristic coals, mills and classifier settings are such as to give a finer product (85% passing 75μm) necessary for complete combustion. Milling energies are higher (up to 40kWh/t). The product size characteristics of coals from vertical spindle mills are shown in Table 3.4. Figure 3.9 shows the different product size distributions for two operating UK coal fired power station boilers (Hill 2000), the average distributions are presented from 6 ball mills and 8 vertical spindle mills used to feed the separate boilers.

Table 3.4 Characteristics of Pulverised Fuel, (Spiers 1950)

Type of coal and degree of fineness.	Grading: % passing British Standards sieve (mesh) number.				Approximate power for grinding kWh/t
	44 (355μm)	60 (250μm)	100 (150μm)	200 (75μm)	
Bituminous coal					
Normal	99		85-88	55-65	12-16
Fine	99.5		88-92	60-70	15-20
Superfine		99.5	95-98	80-90	23-28
Anthracite					
Normal			99-99.5	85-90	30-40

Values based on British coals containing 3% moisture (cold air swept), and 10% moisture (hot air swept)

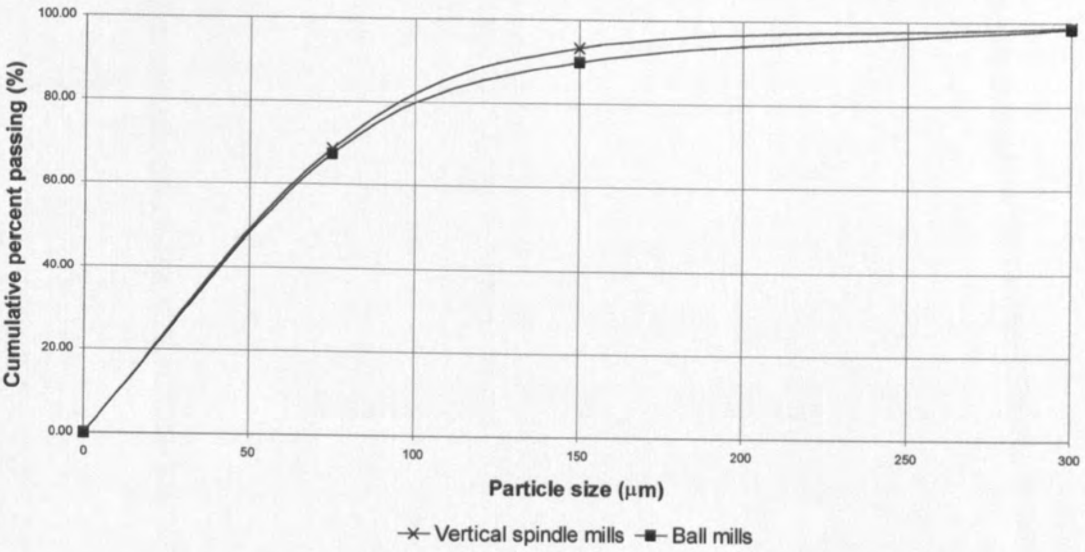


Figure 3.9 Average measured product size distributions from 8 vertical spindle mills and 6 ball mills (Hill 2000)

3.10 Pulverised fuel (PF) production

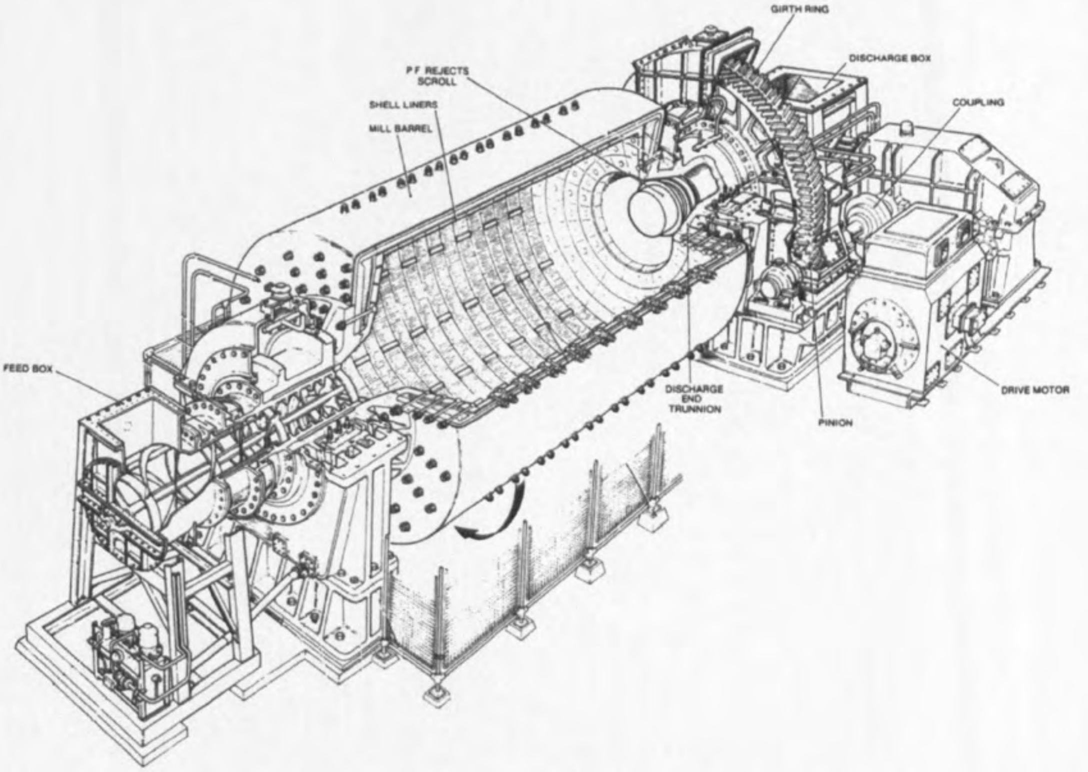


Figure 3.10 Tube ball mill (C.E.G.B. 1971)

The majority of mills in UK power stations are of similar design and based on that of the vertical spindle mill. These are suitable for bituminous coal. For low volatile coals (generally of higher grindability) a tube ball mill may be more appropriate. This mill (Figure 3.10) is also suitable for high and abrasive ash-containing coals. However, in contrast to the vertical spindle mill (Figure 3.12), tube mill performance is more adversely effected by coal moisture content.

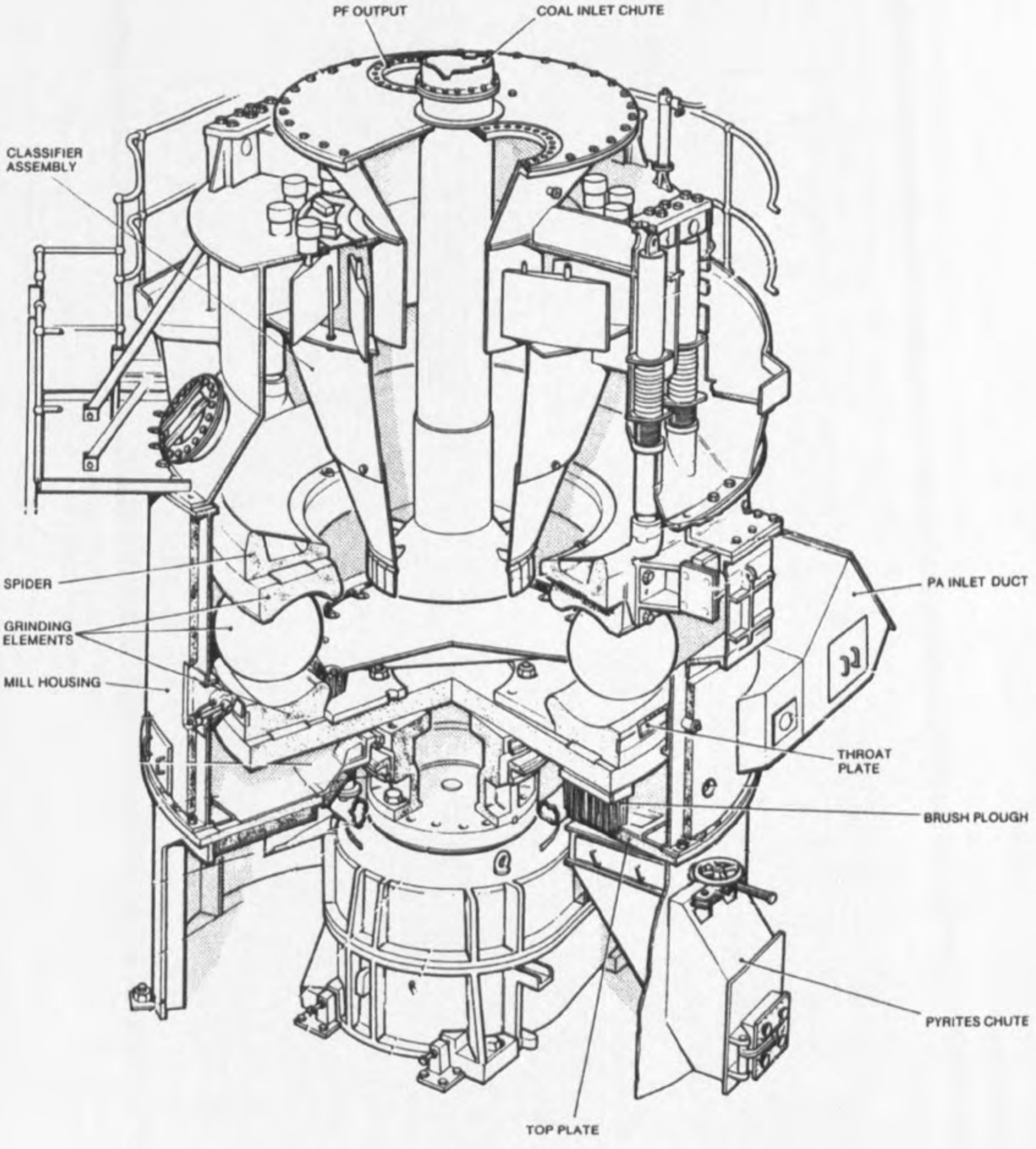


Figure 3.11 10E Vertical spindle mill (C.E.G.B. 1971)

The particular choice of mill depends on coal type, power consumption, maintenance cost and plant layout. Tube ball mills can be operated under suction or pressure. However, exhaust fans (providing induced draught) can suffer from erosion of impeller blades and lining. Mills operating under pressure require efficient trunnion seals to prevent leakage. Heated air is supplied to the fans to dry the coal during milling and the majority of fans are designed to provide sufficient heat at a rate capable of evaporating the whole of the surface moisture and up to half of the inherent moisture of the wettest specified coal. Clearly, this feature must be influenced by the coal charge and gas residence time within the mill. The ball charge is usually rotated at 70% of the 'critical speed' for optimum operation. Tube mills also have the advantage of on-load replacement of the grinding media although, whilst they are usually more reliable than spindle mills, their specific power consumption is often higher than that of the spindle mills for bituminous coals.

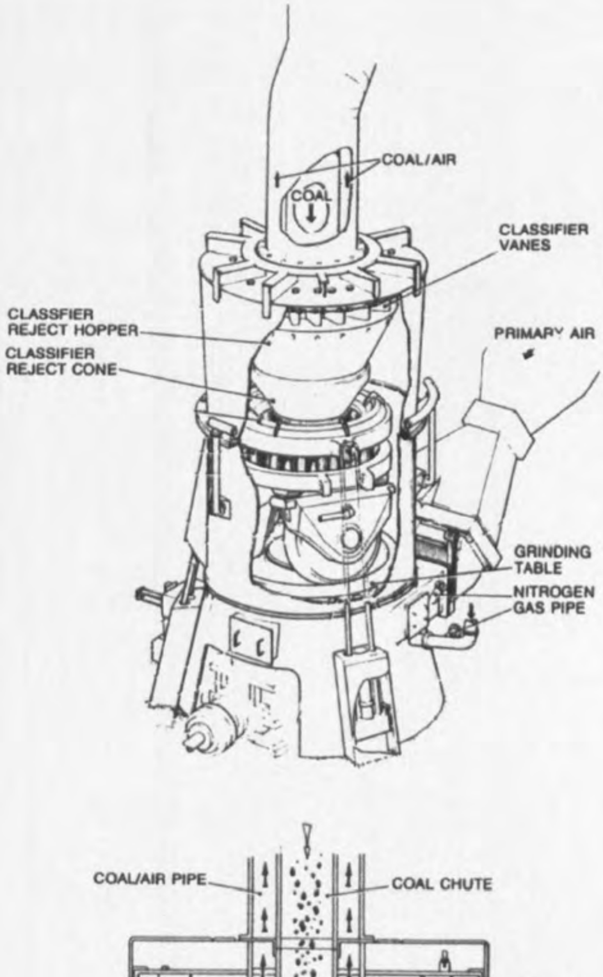


Figure 3.12 PHI Vertical spindle mill (C.E.G.B. 1971)

Vertical spindle mills are hot air-pressure fed (forced draught) and, accordingly, few problems exist with fan erosion. However, irrespective of the grinding element loading (spring or hydraulic) ball and shell wear rates are appreciable and off-line maintenance of these elements is needed. To maintain a symmetrical flame profile in the combustion chamber of corner-fired boilers, the coal to each burner is fed from a number of mills located such that, if any one mill is not in service the (reduced) coal flow remains evenly distributed amongst the four burners.

3.11 Regulations

Regulations relating to the control of gaseous emission levels can differ between countries and legislative limits can depend on the age and location of the plant, economic considerations and on the best available technology.

Currently, the two main international governing bodies monitor sulphur dioxide emissions are: - (i) The European Communities Legislation which deals with the limitation of emissions from large combustion plant, and (ii) The United Nations Economic Commission for Europe (UNECE) convention on long range trans-boundary air pollution. More than thirty countries are signatories to these conventions, including most European countries and North America. Other countries have their own standards and limitations on SO_2 emissions; however, in some cases, the limits are intended only as guidelines.

Table 3.5 European Community sulphur dioxide emission targets (Couch 1995)

Member state	**SO_2 emissions kt/y**	**SO_2 emission target kt/y**			**Percentage reduction from 1980**		
	1980	**1993**	**1998**	**2003**	**1993**	**1998**	**2003**
Belgium	530	318	212	159	-40	-60	-70
Denmark	323	213	141	106	-34	-56	-67
Germany	2225	1335	890	668	-40	-60	-70
France	1910	1146	764	573	-40	-60	-70
Greece	303	320	320	320	+6	+6	+6
Ireland	99	124	124	124	+25	+25	+25
Italy	2450	1800	1500	900	-27	-39	-63
Luxembourg	3	2	1.5	1.5	-40	-50	-50
Netherlands	299	180	120	90	-40	-60	-70
Portugal	115	232	270	206	+102	+135	+79
Spain	2290	2290	1730	1440	0	-24	-37
United Kingdom	3883	3106	2330	1553	-20	-40	-60
EC Total	14438	11066	8403	6141	-20	-42	-57

Table 3.5 shows the European Community's predicted targets for the reduction of SO_2 emissions from large combustion plant (based on measured emissions in 1980) between 1993 and 2003. The standards differ depending upon whether the coal is indigenous or imported and the extent of the country's dependence upon coal. The clear implication is the need to pre-clean (wash) coal prior to combustion and/or incorporate flue gas cleaning measures following combustion of the coals. In some cases, the cost of complying with the limitations has led to the use of other fuels. The United Kingdom, for example, has substantial indigenous coal reserves, yet cheaper imported coals can currently being used for power generation. In some cases, older generating units have been replaced by gas burning units. The legislation sets quotas from plant to plant; in particular, new plants capable of burning high sulphur coals may be required to incorporate gas clean-up capable of removing more than 90% of the sulphur content.

Other countries have stricter limits, the United States of America has a maximum level of 8.9MT/y of SO_2 of which 5.6MT/y is from heavy industrial units. For comparison purposes, the UK target level (for 2003) is 1.55MT/y (Table 3.5). The U.S. system sets allowances with a maximum emission of 3100mg/m^3 per electrical power station (based upon the 110 existing plants producing more than 110MW energy). However, those plants that do not require the full allowance are permitted to share their balance with other plants. Japan, although not a signatory of either convention, operates what is known as the K-value control standard and limits the plant emission levels to an extent dependent on the total air pollution within the localized area.

Regulations are less stringent in China and India where it may be difficult to fund the modifications and retrofit older coal-fired plants. However, it is anticipated that newer plant will be required to operate at lower levels of SO_2. The majority of other developing countries could generally import coal and hence choose those of low sulphur content provided they were affordable.

At present, other emissions are subject to few constraints. However, legislation may be introduced in the future depending on the outcome of research into the effect of other air toxins. There has already been mounting pressure to reduce NO_x emission levels from power stations by modification of the combustion process e.g. staged air injection into the boiler. Such modifications would be cheaper and easier to install than those for the reduction in SO_2 emissions.

With regard to discharge from power station plants, strict water run-off constraints are usually introduced by local sewage and water authorities in order to limit the volume and concentration of waste effluents to ground water supplies and rivers. Regulations on solid residues again are dealt with locally and generally depend on coal ash content. The common aim is to reduce soil contamination and any possible effects of natural leaching of heavy metals to ground water.

3.12 Future coal processing technology

Currently, there are three areas of major research worldwide viz methods of producing clean coal for emission reduction, improving the thermal (cycle) efficiency for coal combustion in power plant and the use of coal bed gasification.

Emissions control research involves the removal or reduction of toxic byproducts generated during the combustion of coal. Due to legislation and 'capping' policies imposed by governments there is a requirement for technologies to control nitrates, sulphurous compounds and green house gas (water vapour, carbon dioxide, methane and nitrous oxide) emissions. Current developments in this field include flue gas desulphurisation by means of a wet-lime or limestone process and low-NO_x burners which delay the mixing of air and coal in a manner such that the capabilities of producing nitrates is reduced.

Currently, coal-fired power station boilers use pulverised fuel for firing and operate at thermal efficiencies limited by steaming conditions to 41% for the most modern designs (the limiting feature arises from the steam to turbine energy transfer rather than heat transfer efficiency in the boiler). More recent developments are based on supercritical steam cycles which give increased the thermal efficiencies of up to 45% using high-pressure superheated steam cycles. The reliability of these plants has been proven and they are commercially available; plant designs currently in research and development stages show promise of thermal efficiencies above 50%. In particular, Integrated Gasification Combined Cycle (IGCC) power generation is based on the gasification of coal with the gas expanded subsequently into a gas turbine some 67% of the power is generated in this way with the remainder being generated by conventional heat transfer steam generation. Studies of hybrid cycles (gasification and combustion) in the USA, Germany and the UK are at the research stage. The process uses high-pressure steam and air for gasification of the coal (~80% of the fuel) and the remaining char is burnt as a single fuel. Both gas turbine and steam-driven turbines are used.

The development of coal bed gasification is based on the theory of producing methane from coal beds by direct injection of oxygen and water into the bed (The thermal efficiency of natural gas is considerably higher-50-56% total efficiency-than that of pulverised fuel coal combustion). Other advantages include little to no mining, ease of handleability of the fuel and possible reductions in process energy requirements (milling, coal processing). This may possibly be utilised on remaining coal commodities in redundant mines (D.T.I. 1999).

CHAPTER FOUR

REVIEW OF MICROWAVE STUDIES UPON COMMINUTION

4.1 Introduction-Review of microwave studies upon comminution

This section provides a background to the grindability of materials and conventional comminution theories. In particular, the effect of heat treatment to reduce the resistance of a solid material to size reduction and the relative affect of microwave and convective heating are reviewed.

4.2 Breakage theories

Whatever the reason for size reduction, whether comminution is necessary to liberate valuable minerals from a host rock or to produce pulverised fuel from coal, the principles of comminution are similar. Inter-atomic bonds are only effective over small distances and can only be broken if sufficient energy is available. Many size reduction mechanisms produce compressive or tensile stresses to generate the required force for breakage. Breakage however, does not only depend upon the applied stress, each material has unique structures that resist breakage. Natural materials (e.g. ores) commonly contain a number of mineral species dispersed as grains of various sizes that are unevenly distributed. The distribution of the applied force is then dependant upon the individual mechanical properties of the inherent minerals and upon weaknesses (flaws) within the matrix. Cracks act as stress concentration points, the stress at such points depending upon the crack length and beyond a certain distance the force may be sufficient to break the atomic structure further. Such ruptures will increase the crack length and subsequently increase the stress concentration leading to rapid crack propagation and particle fracture (Wills 1992).

Over the years, common comminution theories have been proposed and material tests developed in order to further the understanding of material breakage.

4.2.1 Comminution theory

Comminution theory is concerned with the relationship between energy input and the product particle size made from a given feed size (Bond 1961).

In 1867, the first comminution theory (proposed by Von Rittinger) stated that the area of new surface produced is directly proportional to the useful energy consumed in size reduction. Rittinger's law is concerned with the relationship between the surface area of one tonne of particles of uniform diameter '*d*' and the work input per ton, both of which are considered to be proportional to 1/*d* (Bond 1961). Rittinger's law equates to Equation 4.1:

$$E_{nergy} = K\left(\frac{1}{D_2} - \frac{1}{D_1}\right) \qquad (4.1)$$

E_{nergy}	Energy input (kWh/t)
K	Constant dependent upon material
D_1	Initial particle diameter (m)
D_2	Final particle diameter (m)

A second theory of comminution, proposed by Kick (1885), states that the work required for breakage is proportional to the size reduction of the material. In particular, the reduction ratio (*Rr*) of the feed particle size (F) to the product particle size (P) is related to the work input per tonne (*W*) as shown in Equation 4.2 (Bond 1952).

$$W = \log Rr / \log 2 \tag{4.2}$$

W Work input per tonne (kWh/t)
Rr Reduction ration (F/P)

Both theories are considered as inappropriate as they do not conform to commercial crushing and grinding practice. The current accepted theory is that of Bond's third theory of comminution developed in 1951 (Bond 1961). However, it has also been suggested that, the relationship between the particle size and breakage energy is a composite of the three theories, a material may comply with all three theories to an extent dependant upon particle size (Lowiston, 1974). It is reasoned that grinding obeys Kick's law if the particle is greater than 10mm diameter, Bond's theory is satisfactory if the particle size range is between 10mm and 100μm (considered the size range for conventional rod and ball milling) and that Rittinger's law is applicable for fine grinding 1mm – 10μm.

4.2.2 Bond Work Index (Wi)

The Bond Work Index is based on the theory that the work input is proportional to the new crack tip length produced during breakage taking into consideration the original feed size of the material. The Work Index is a parameter that expresses the resistance to grinding of that material and is numerically defined as the specific energy (kWh/t) required to reduce that material from infinite size to 80% passing 100μm. Bond's third theory is generally expressed in the form:

$$W = \frac{10Wi}{\sqrt{P}} - \frac{10Wi}{\sqrt{F}} \tag{4.3}$$

W	Work input (kWh/t)
F	80% passing sizes of the feed (μm)
P	80% passing sizes of the product (μm)
Wi	Bond Work Index (kWh/t)

4.2.3 Relationships between Bond Work Index and Hardgrove Grindability Index

As previously stated (Chapter 3.4), the Hardgrove Gindability Index is a standard test method used in the coal industry. The Hardgrove Grindability Index, however, is limited in use and is a determination of the relative grinding behaviour (in a ring ball mill) as a function of coal rank rather than for scale up design applications, as may be achieved from a knowledge of the Bond Work Index.

A correlation between Bond Work Index (Wi) and Hardgrove Grindability Index (HGI) was proposed by Bond in 1954 (Equation 4.4) and subsequently, a revised version was published in 1961 (Equation 4.5) (Bond 1961).

$$Wi = \frac{88}{\mathrm{HGI}^{0.3}} \tag{4.4}$$

Wi	Bond Work Index (kWh/t)
HGI	Hardgrove Grindability Index

$$Wi = \frac{435}{\mathrm{HGI}^{0.91}} \tag{4.5}$$

Further work by McIntyre and Plitt (1980) led to an evaluation of the above two relationships which led to that shown in Equation 4.6 (McIntyre 1980):

$$Wi = \frac{1622}{\mathrm{HGI}^{1.08}} \quad \text{For } Wi > 8.5 \tag{4.6}$$

To verify which of these relationships was the most appropriate for correlation between Bond and Hardgrove indices, both the standard Hardgrove gindability test (BS 1016, 1981) and the Bond Work Index were experimentally determined using a British coal (P-2 coal). The Hardgrove Grindability Index was determined by the Coal Research Establishment Group. The measured Hardgrove Grindability Index of this coal (HGI = 53) was correlated to the Bond Work Index from Equation 4.4, 4.5, and 4.6 respectively (Table 4.1).

Table 4.1 Calculated Bond Work Index from the measured value of the Hardgrove Grindability Index

Proposed by:	Equation	Formula	*Wi* (kWh/t)
Bond 1954	3.4	$Wi = 88/(HGI^{0.3})$	26.7
Bond 1962	3.5	$Wi = 435/(HGI^{0.91})$	11.7
McIntyre and Plitt 1980	3.6	$Wi = 1622/(HGI^{1.08})$	22.3

Results (Table 4.1) show considerable variance in *Wi* from these correlation's. A Bond test was carried out on a 30kg sample of P-2 coal as described in Appendix A. The *Wi* was determined at 75μm and 106μm cut sizes and the standard Bond Work Index calculated from the composite curve (Figure 4.1).

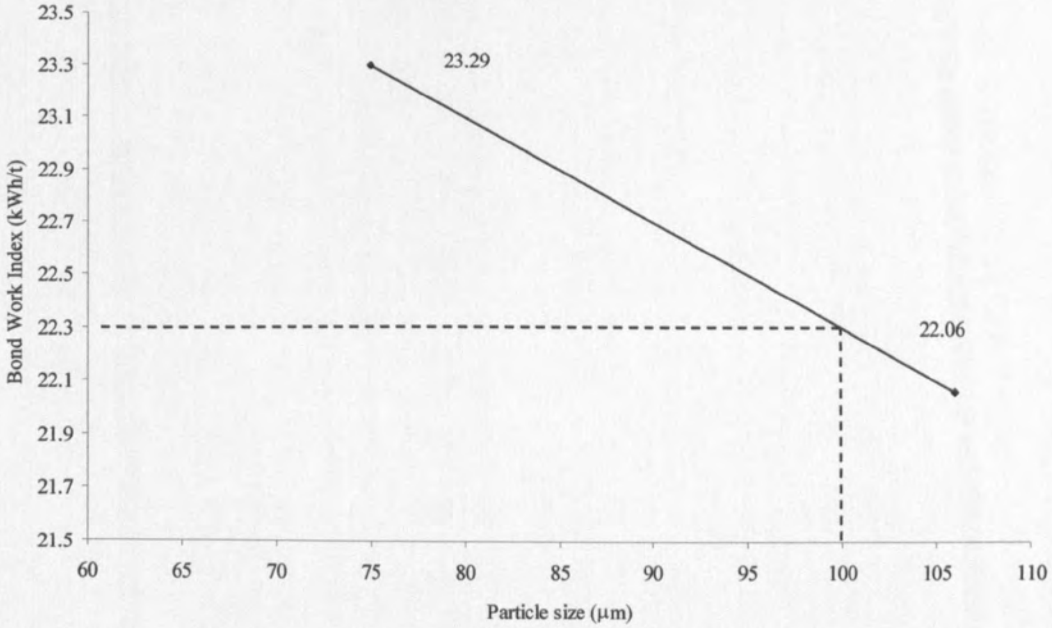


Figure 4.1 Determination of Bond Work Index for P-2 coal

Figure 4.1 shows that the experimentally determined *Wi* (100μm) for P-2 coal is 22.3 kWh/t, this is similar to the value derived from the relationship proposed by McIntyre and Plitt (1980).

If it is then assumed that the correlation expressed in Equation 4.6 is the most suitable, then interrelationships can be applied on coals of HGI >160 or *Wi* <8.5.

4.2.4 The Relative Work Index (RWI)

The Relative Work Index (derived from the Bond Work Index) is simply a method to compare the 80% passing size of the milled product to that of a reference material.

Due to the time required to undertake the standard Bond test Berry and Bruce (1966) developed a comparative method to determine the Work Index of an unknown material. The method requires the use of a reference material of known Work Index. An identical weight of both samples are ground within a mill at constant speed so that the power consumption is equal and can be used as the common term in a derivation of simultaneous equations ($Wr=Wt$). If '*r*' denotes the reference material and '*t*' the material under test, using simultaneous equations as expressed in Equation 4.3, the Work Index of the test material can be determined (Equation 4.7).

$$Wr = Wt = \frac{10Wir}{\sqrt{Pr}} - \frac{10Wir}{\sqrt{Fr}} = \frac{10Wit}{\sqrt{Pt}} - \frac{10Wit}{\sqrt{Ft}} \tag{4.7}$$

W	Work input (kWh/t)
F	80% passing sizes of the feed (μm)
P	80% passing sizes of the product (μm)
Wi	Bond Work Index (kWh/t)

For material '*r*' and '*t*' respectively.

Rearranging these equations gives the Comparative Work Index (CWI):

$$CWI = \frac{Wit}{Wir} = \frac{\dfrac{10}{\sqrt{Pr}} - \dfrac{10}{\sqrt{Fr}}}{\dfrac{10}{\sqrt{Pt}} - \dfrac{10}{\sqrt{Ft}}} \tag{4.8}$$

The equation can be converted, exclusive of the Bond Work Index of either material, to provide a Relative Work Index i.e. relative to the reference material and expressed on a percentage scale (Equation 4.9). This is similar in methodology to the relative grindability scale used by Hardgrove to compare the grindability of coals and represents a simple way to ascertain whether the grindability of one material is greater than another.

Within the current study, the non-treated coal sample is classed as the reference sample and the treated samples as the test samples, thus giving a comparison between milled coal products of both untreated and microwave treated coals.

$$\mathrm{RWI} = \frac{\dfrac{10}{\sqrt{Mn}} - \dfrac{10}{\sqrt{Fn}}}{\dfrac{10}{\sqrt{Mt}} - \dfrac{10}{\sqrt{Ft}}} \times 100 \qquad (4.9)$$

RWI	Relative Work Index (%)
Fn	80% passing size of the feed coal, non-treated coal (μm)
Mn	80% passing size of the milled coal, non-treated coal (μm)
Ft	80% passing size of the feed coal, microwave treated coal (μm)
Mt	80% passing size of the milled coal, microwave treated coal (μm)

4.3 Affect of heat treatment on grindability

The effect of heat treatment on grindability has been studied since the beginning of the 20th century. In particular, ore grindability has been investigated due to the complex mixtures of minerals within a host ore body. Fractures have been reported as a result of heat treatment alone and in some cases, these fractures were responsible for a reduction in the Work Index and gave improved mineral liberation. The following sections describe previous research concerning thermally-assisted liberation, microwave properties in relation to mineral heating and their effect on microwave-assisted comminution.

4.3.1 Thermal assisted Liberation

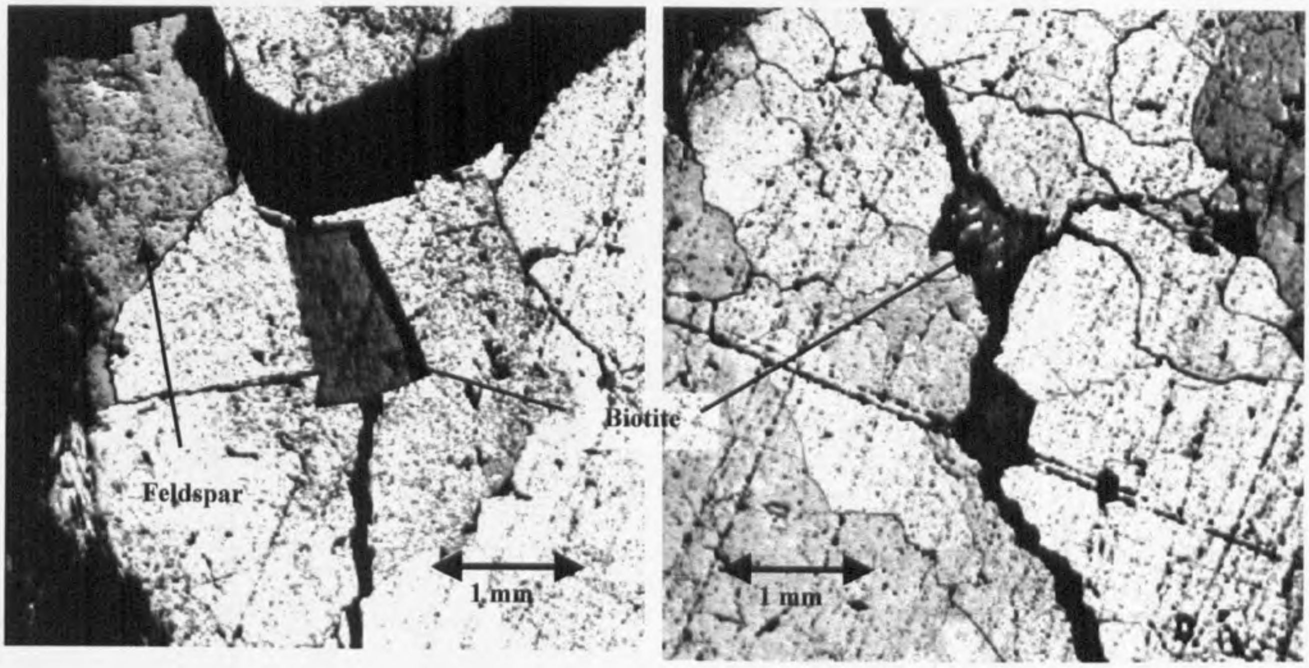


Figure 4.2 Intergranular cracking (Young 1997)

Thermally-assisted liberation is concerned with rock fracture characteristics upon heating. Comminution is the most energy intensive process within mineral processing and up to 70% of the total energy requirements of some metal extractions is used in grinding (Walkiewicz 1991). As early as 1918, a study of the application of heat energy to reduce grinding resistance was carried out with the ultimate aim to reduce comminution energy (Yates 1918, Holman 1927). Typically, ores contain a number of minerals of different mechanical and thermal properties. Each mineral has different thermal expansion properties, which upon heating and cooling create stresses, which may lead to localised fractures. Intergranular and transgranular fractures can be produced as a result of heat treatment. Figure 4.2 shows an intergranular fracture within a granite (consisting primarily of quartz, feldspar and biotite) after heat treatment within a muffle furnace at 650°C. Fractures may have been produced because of the α to β phase change of quartz at 573°C resulting in a volumetric expansion of 0.86% (Young 1997). Thermally-assisted liberation research by Fitzgibbon and Veasey (1990) reported significant reductions in grinding resistance could be achieved. However, the benefits of heat treatment were uneconomical on tested ore species.

4.4 Microwave assisted comminution

Thermally-assisted liberation to date is generally considered as uneconomical due to the high energy requirements for bulk heating. However, microwave energy presents an opportunity to reduce the overall energy requirements of thermally-assisted liberation in so far as it provides an in-situ method of heating in contrast to conventional heating, which relies on heat transfer by conduction. Furthermore, since not all materials absorb microwave energy, differential heating and expansion could be produced without bulk heating.

Subsequent sections review topics related to microwave-assisted comminution and include the relationship between dielectric properties, heating rates and their effect on grindability.

4.4.1 Dielectric heating rates

Early research concerning the effect of microwave energy on natural materials showed that all materials heat at different rates. As with thermally-assisted liberation of minerals from ore bodies, expansion rates may induce fracture propagation. Chen et al (1984) reported the relative transparency of some minerals to microwave radiation with varying power at a frequency of 2.45GHz. The study concluded by grouping the test minerals, i.e. group 1 minerals which exhibit poor heating and relative transparency (most silicates, carbonates, sulphates and some oxides and sulphides) and group 2 minerals which show good heating characteristics (most sulphides, arsenides, sulphosalts and sulphoarsenides). Following this study, Walkiewicz et al (1988) produced additional data for mineral heating at a power of 1kW and a frequency of 2.45GHz. Results indicated that metal oxides and sulphides heat rapidly, most metals generally reflect microwave radiation, most chloride minerals are poor receptors and common gangue minerals (silicates) are relatively transparent to microwave radiation. The study also showed that intergranular and transgranular fractures may result in ore bodies when exposed to microwave radiation. Similar conclusions have been made by other researchers (Dobson 1991, Worner 1989, Harrison 1995). Further work by McGill et al (1988) indicates that microwave power has a significant effect upon heating rates, heating rates increase for receptive minerals. In contrast, low loss minerals showed lower heating rates.

4.4.2 Dielectric properties of minerals

Heating rates are dependent upon the material dielectric properties and those for mineral species has been reported at frequencies between 300Hz and 1000Hz (Church 1988). The report showed that minerals with high dielectric constants (and loss factors) have higher heating rates than minerals of lower dielectric properties. Each mineral tested had unique dielectric properties. However, in general, oxides, tungstates and some carbonates had high dielectric constants ($25>\varepsilon'>6$), whilst most silicates had much lower values ($\varepsilon'<6$). Nelson et al (1989) carried out a study on metal oxides and silicates at frequencies of 1.0, 2.45, 5.5, 11.7, 22.0GHz and concluded that, whilst dielectric constants decrease with increasing frequency, a resonance frequency (optimum frequency unique to material) may exist within the microwave frequency range.

4.4.3 Microwave exposure of minerals and comminution behaviour

As different thermal expansion coefficient exist between different minerals, localised fractures may be produced upon heating. Thermally-assisted liberation using microwave energy has been studied by a number of researchers since the first publication "Microwave Assisted Grinding" by Walkiewicz in 1991. Similar principles apply for thermally-assisted liberation. However, microwave energy, depending upon the ore body, may only be absorbed in part by the ore (transparency of some mineral species), reducing the energy requirements when compared to conventional furnace heated thermally-assisted liberation.

A reduction in the Bond Work Index of between 10% and 24% was reported by Walkiewicz et al (1991) after microwave heating (at a power of 3kW) of iron ore samples. Harrison et al (1996) investigated the effect of microwave radiation on the grindability of a number of different minerals and coals (The effect of microwave radiation on coal grindability is discussed in Chapter 5). Samples of each mineral were exposed to microwave radiation at a power of 0.65kW for five minutes. Minerals having low heating rates exhibited no change in grindability, minerals with medium and high heating rates showed increases in surface area after microwave exposure and ensuing improvements in grindability. Minerals such as pyrite and barytes are reported to have reductions in Relative Work Index (RWI) of 35% to 40% respectively, whilst the greatest improvements in RWI (more than 70% reduction) were shown for bornite and chalcopyrite (Table 4.2). Minerals that have the ability to absorb microwave radiation at high heating rates generally showed subsequent changes in comminution behaviour (Harrison 1996).

Table 4.2 Microwave grindability and heating rates of selected minerals (Harrison 1996)

Mineral	Measured heating rate (0.65kW, 2.45GHz) (°C/s)	Relative Work Index (300s irradiation at 0.65kW, 2.45GHz) milled for 45 minutes (%)
Quartz	0.07	100
Feldspar	0.1	99
Barytes	0.09	59
Bornite	2.23	22
Chalcopyrite	2.21	29
Pyrite	1.89	62
Wolframite	0.09	27
Hematite	0.81	47

Thermal expansion and intergranular fracture along the grain boundaries of a single pyrite particle within a calcite host rock was examined using a finite element numerical model developed by Salsman et al (1995). The study predicted significant differential heating using a short pulse microwave source; the associated thermomechanical stresses suggested a weakening of the host rock and reduction in Work Index (Salsman 1995).

Kingman and Rowson (1996) have considered the effect of microwave energy on a number of minerals. Reduction in Bond Work Index and Ring-Loaded strength after microwave exposure at powers of 1.3kW and 2.6kW were reported upon massive Norwegian ilmenite ore and Palabora carbonatite. Further studies were directed towards evaluating the potential of microwave energy on the down stream processes- in particular the leaching of copper and gold ores after irradiation.

Previous research has demonstrated the potential capabilities of improved grindability by microwave energy upon particular minerals, depending upon their dielectric properties, their position within an ore structure and the applied microwave field properties. It is noted, however, that previous publications have considered the economics of such microwave-assisted grinding unfavourably. Clearly, further research is necessary to optimise process conditions and to assess the effect of material behaviour upon microwave exposure.

CHAPTER FIVE

THE EFFECT OF MICROWAVE RADIATION ON COAL

5.1 Introduction-The effect of microwave radiation on coal

Much research has been carried out into the effect of microwave radiation on coal with emphasis given to assessing the changes in coal characteristics following irradiation- in particular, dielectric properties, desulphurisation and grindability.

5.2 Dielectric properties and microwave heating rates of coal

Investigations concerning the dielectric properties of individual minerals found in coals show significant differences in absorption characteristics and subsequent differential heating (Florek 1996, Batt 1992, Harrison 1996) (Chapter 4). More specifically the dielectric properties of coal depend on a range of factors including coal rank, moisture content, mineral composition, temperature and the frequency of microwave radiation. The wide range in variables make measurements by various workers difficult to collaborate (Yang 1987). For example, such studies show that the relative dielectric constant of Chinese coal from different origins varies from 3.22 to 4.57 at an operating frequency of 2.45GHz. The report also highlights the subsequent change in favour of microwave heating through dielectric property measurement (increasing dielectric constants) with additions of caustic solutions.

Unlike some minerals, the dielectric constant of coal has been shown to decrease with increasing temperature (Balanius 1981). The workers also determined the dielectric constants of a range of bituminous coals over the frequency range 8.2GHz to 12.4GHz and for varying particle sizes. No significant changes were reported in the measured dielectric constants over the latter frequency range. However, coarser coal particles showed an increase in the dielectric constants (Balanius 1976). A study of dielectric properties within the frequency range 0.1 – 6GHz showed decreasing values of dielectric constant and loss factors from 0.1GHz to approximately 4.5GHz (Figure 5.1 and 5.2) (Harrison 1997).

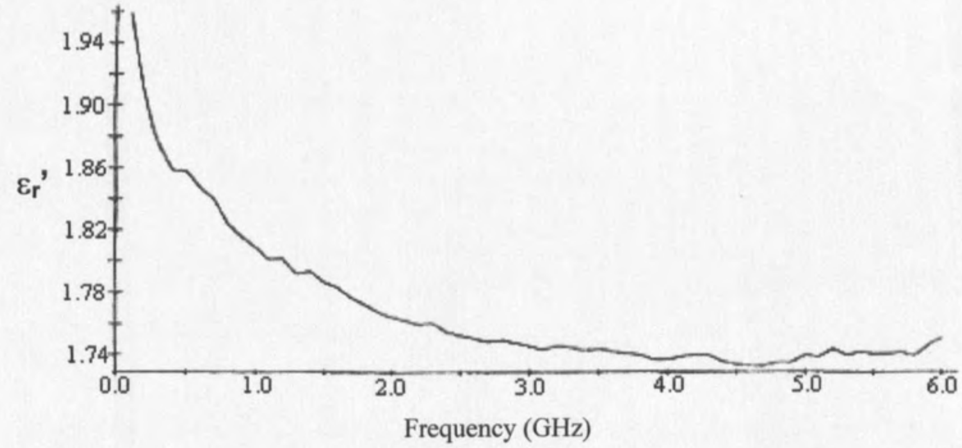


Figure 5.1 Dielectric constant (ε_r') of coal between the frequency range 0.1 – 6GHz (Harrison 1997)

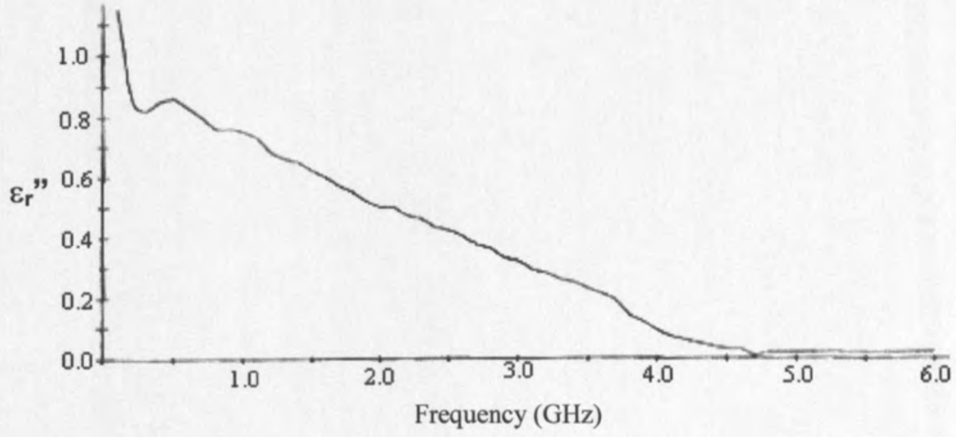


Figure 5.2 Loss factor (ε_r") of coal between the frequency range 0.1 – 6GHz (Harrison 1997)

Dielectric measurements show that coals can be rapidly heated within a microwave field and the heating processes can be to depend on time, input power and frequency. Table 5.1 shows the relative difference between the dielectric constants for a dry coal, pyrite and ash. It is inferred that pyrite can be heated relatively rapidly due to its associated high absorbency (Chatterjee 1991).

Table 5.1 Measured dielectric properties utilised in the numerical models (frequency 2.45GHz) (Chatterjee and Misra 1991)

	Relative Dielectric constant ε_r'	Conductivity O_c (S/m)
Dry coal	3.0	0.061
Pyrite	7.0	1.04
Ash	4.6	0.0

To account for the number of variable components within a given coal, mixture models can be used to determine an average complex permittivity (Masayuki 1995). Such procedures have also been employed to predict the permittivities associated with the density difference arising from mineral inclusions and measured particle sizes (Nelson 1996, Andrzej 1999).

Knowledge concerning the dielectric constants of low rank coals is limited and it is possible that such coals may be highly receptive to microwave heating due to their increased moisture content. Further work is required to identify the most suitable coals for commercial applications.

5.3 Microwave drying of coal

"Here is a way to put most of the drying energy into where you want it-to the moisture within the coal particles rather than to the particle's entire body"(Chironis 1986).

Preferential heating of water in coal has led to investigations regarding the use of microwaves in coal drying applications. The moisture content of fine coal is often high and can create problems in bulk handling when coal is blended, conveyed or stored; more over, when coal has a low heating efficiency. Whilst it has been claimed that conventional thermal drying (by means of hot-air) can be efficient, it may not always be suitable for fine coals containing a high moisture content. The ensuing coal can often result in dust problems (Bates 1989). Chironis et al has shown that microwave drying efficiencies can reach 97% for some coal fines (<6.35mm). Similar research by Lindroth et al (1986) showed drying efficiencies (63%-90%) considerably greater than those achieved using conventional thermal drying techniques. Standish et al (1987) reported the rate of moisture removal by microwave energy in some brown coals was approximately twice that for conventional drying. Dielectric modeling techniques have been used to predict heating rates and suggest that at a frequency of 2.45GHz and at a power of 0.663kW coal samples can reach elevated temperatures sufficient to remove moisture (Chatterjee 1991).

Microwave drying techniques can also be used as a method of moisture content determination comparable to the standard ASTM proximate analysis (Jacobs 1984). Laboratory and pilot-scale trials have been used to investigate the potential for online moisture analysis. Many researchers have studied within this field and have claimed accuracies of ± 0.5% on continuous coal flow moisture analysis (Kalinski 1978, Hall 1972, Cutmore 1987).

Research demonstrated that the reduced coal drying times could be achieved without coal degradation in the form of ignition or volatile matter loss. On-line moisture determination using microwave energy has also shown to be a rapid and accurate method especially for medium, high rank coals; however, to date, no commercial on-line system is available.

5.4 Microwave pyrolysis of coal

Research has been carried out to establish whether microwave radiation (as opposed to conventional heating) can be used for coal pyrolysis (Larry 1986, Gasner 1986). However, it was concluded that microwave energy was not effective without the use of plasma initiation. The purpose of the study was to determine if a higher percentage of liquid products could be achieved using microwave energy as opposed to heating in a muffle furnace. Copper wires were employed as the plasma source. Both conventional and microwave heating generated similar quantities of liquid product. However, low power microwave radiation yielded products at a much lower residence time and favoured tar production (Table 5.2).

Table 5.2 Comparison of product yields obtained by microwave and convective heating processing (Larry et al 1986)

Product yield, mass %		
	Microwave	Convection
Gas	17	30
Liquid	14	1
Char	69	69

Monsef-Mirzai et al (1995) reports the difference in tar production using copper oxide, iron oxide and coke as microwave receptors (plasma initiation source) in Nitrogen flow microwave pyrolysis. Tar products of up to 49wt% are achieved using CuO, 27wt% with Fe_3O_4 and 20wt% with metallurgical coke. There was ensuing discussion concerning the possibility of continuous processing using fluidised bed technology with associated rapid microwave pyrolysis capability.

5.5 Microwave desulphurisation and demineralisation of coal

Various authors (Zavitanos 1981, Kelland 1988, Viswananthan 1990, Butcher 1995) have suggested the use of microwave radiation for the desulphurisation of coal. Environmental legislation imposes a limit to the sulphur release into the atmosphere. This has led to the use of flue gas desulphurisation equipment and expensive removal options such as flotation, it is also a significant factor influencing selective coal blending (reduce total sulphur content by blending with a low sulphur coal). References suggest that it may be possible to use microwave energy to remove part of the sulphur bearing material from some coals prior to combustion. In principle, two routes are possible.

(i) Conversion of pyrite to pyrrohotite followed by magnetic separation.

(ii) Preferential dissolution of sulphur by molten caustic solutions.

Microwave radiation has also been applied to coals wetted in an acid or caustic solution to dissolve inorganic (mineral) coal components as a means of coal cleaning.

5.5.1 Microwave conversion and separation of pyrite

Considerable research has been carried out concerning the partial oxidation of pyrite to liberate sulphur from coal. When heated to temperatures of, typically, 300°C Pyrite (FeS_2) can phase to a form similar to that of mineral pyrrohotite (FeS) with the release of sulphurous gases (predominantly sulphur dioxide and hyrogen sulphide). In particular, as stated previously (Section 5.2) pyrite in coal heats at a rate of between 2 and 10 times that of the parent coal (Zavitanos 1981). The properties of oxidised pyrite significantly change and it is claimed that the magnetic susceptibility greatly increases. Experiments have demonstrated that, if only 1% of the total mass of pyrite is decomposed, the magnetic susceptibility can increases of the order of two magnitudes (Bluhm 1980, Butcher 1995). Butcher (1995) investigated the feasibility of magnetic separation of enhanced magnetic pyrite and concluded that low intensity magnetic separation and any wet magnetic separation methods would not be effective due to high calorific value losses. Butcher (1995) did, however, suggest that optimum particle size ranges exist which significantly improve the microwave enhanced magnetic properties of pyrite and its subsequent liberation. Experiments have shown that up to 80% of the pyritic sulphur can be removed using a high gradient semi conductor magnetic separator (15T) (Kelland 1988). Other workers (Zavitanos 1981, Viswananthan 1990) provide supporting experimental evidence using high gradient magnetic separators.

5.5.2 Microwave desulphurisation of coal using caustic solutions

Since 1979, there have been substantial investigations into the role of caustic alkali solutions (NaOH and KOH) in the microwave desulphurisation of coals. Zavitanos (1979) referred to the removal of pyritic and organic sulphur from coals using alkali caustics; such solutions were shown to be good absorbers of microwave radiation. Due to the selective heating of the caustics in sulphur-rich areas; sulphur-bearing compounds can readily decompose to water-soluble sulphates and acids that can be easily removed by washing. Microwave irradiation of coal, in the presence of sodium hydroxide (16%) and water (10-20%) gave encouraging results with a low loss of volatile matter, a high reduction in pyritic sulphur (90%) and a reasonable reduction in organic sulphur (Yang 1987). The tests also gave an indication of the problems associated with microwave energy distribution in so far as only approximately 3% of the total microwave energy was required for sulphur reduction. Furthermore, an economic evaluation of the results showed promise in comparison with data provided from other desulphurisation methods.

Further research has continued with a variety of aims including whether there is an optimum particle size for pyrite decomposition, determination of suitable microwave operating conditions and the use of molten caustics. With the use of a low gradient magnetic separator, a 70% separation of the total sulphur content was measured after only one minute of microwave exposure (0.5kW, 2.45GHz) with the addition of a molten caustic solution to the test coal (Rowson 1990). Similar work (Butcher 1995) shows that sulphur reduction can be achieved irrespective of particle size. However, particle sizes ranging between 1mm and 0.5mm irradiated for 60 and 90 seconds produced greatest reductions (0.65kW, 2.45GHz). Further suggestions were made regarding the presence of magnetic layers (Fe_3O_4) coating the pyritic inclusions that facilitate magnetic separation (Butcher 1995). Hayashi et al (1990) investigated the potential role of molten caustics in microwave desulphurisation of coals and showed that substantial differential heating could be achieved with coals of low Pyrite content. Total sulphur reductions of up to 60% were achieved and the suggestion was made that the rate-determining feature was mass transfer from the molten caustic into the coal structure.

Other chemical microwave desulphurisation methods have been suggested based on the use of acidic solutions (Andres 1996). Hydrochloric acid was used as a desulphurising agent and, following an appropriate mixing time of twenty minutes, the sulphate and pyritic sulphur could be completely removed in addition to 70% of the organic sulphur.

5.5.3 Demineralisation of coals with microwave energy

Strong alkali and acid solutions have been used to dissolve inorganic minerals within coal. In particular, the application of hydrofluoric acid mixtures have been shown to produce rapid dissolution of nearly 25 elements including: aluminium, silica, zinc, calcium and iron using microwave energy (0.65kW at 2.45GHz frequency) (Nadkarni 1984).

Butcher and Rowson (1995) carried out tests using sodium hydroxide with encouraging results (Table 5.3). However, the latter tests were carried out on coal of –500μm+300μm size range, Nadkarni (1990) suggests that the most satisfactory results were achieved with finer particles (<200μm).

Table 5.3 Ash reductions after microwave NaOH pretreatment (Butcher and Rowson 1995)

Microwave (0.65kW) (2.45GHz) pretreatment time (s)	Feed Ash (%)	Magnetic product Ash (%)	Non-magnetic product Ash (%)	Overall reduction Ash (%)
30	30.47	38.88	21.38	29.18
60	30.47	37.83	25.57	16.07
90	30.47	38.32	26.73	12.27

Even though potential applications of microwave demineralisation are encouraging, associated corrosion problems with containment may prevent commercialisation.

5.6 The influence of microwave radiation on coal grindability

Comminution is an energy intensive and inefficient process and the major part of the energy supplied for grinding is absorbed by the mill with only a fraction of the energy directly utilised in breakage (Herbst 1981). Microwave irradiation has been used to alter the comminution behaviour of coal. Viswanathan (1990) applied microwave radiation at a power of 0.5kW and a frequency of 2.45GHz to 150g samples of a UK coking coal and a non-coking coal each for a duration of one minute. An increase in product size range of 31% and 10% was shown for the microwave treated coking and non-coking coals respectively, implying a significant improvement in coal grindability due to microwave exposure.

Harrison (1997) has carried out substantial work using coal supplied from National Power's West Burton Power Station. The grindability characteristics of three coal blends were measured before and after microwave exposure. To this end, representative samples (500g) were prepared and exposed to microwave radiation at a power of 0.65kW and a frequency of 2.45GHz. The average temperatures of these samples were recorded immediately after irradiation and the samples were ground for 45 minutes in an appropriately-sized rod mill. The coal size distributions were evaluated after 5, 20 and 45 minutes of milling and the data reported as Relative Work indices with regard to that of the non-treated milled sample (Table 5.4).

Table 5.4 The Relative Work Index of sub-bituminous coals with increasing microwave exposures (0.65kW, 2.45GHz)(Harrison 1997)

	Relative Work Index %, ground for:		
No2 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 1 min	92	95	93
Exposure 3 min	85	75	91
Exposure 5 min	84	68	69
Exposure 8 min	81	73	68
No5 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 5 min	85	75	83
Exposure 10 min	84	77	81
No6 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 5 min	96	76	88
Exposure 7 min	88	77	71

Results show that significant improvements in grindability were achieved after 5 minutes exposure. After 8 minutes exposure (No2 coal), 10 minutes (No5 coal) and 7 minutes exposure (No6 coal) microwave treatment was terminated due to coal ignition. As shown in Table 5.5 further work examined the use of an increased microwave power (1.5kW) to reduce the exposure time; however, localised hot spots were observed and experiments terminated after 1, 2 and 3 minutes exposure for No 2, No 5 and No 6 coal, respectively. Harrison (1997) claims that comparable grindability improvements could be achieved with both 0.65kW and 1.5kW microwave power exposures were it not for the pyritic inclusions causing ignition. The work also showed that, with an increase in power the heating rates of both the coal and pyrite are increased. Clearly, pyrite could reach ignition temperature rapidly before any appreciable effect on coal comminution characteristics became apparent.

Table 5.5 The Relative Work Index of sub-bituminous coals with increasing microwave exposures (1.5kW, 2.45GHz)(Harrison 1997)

	Relative Work Index %, ground for:		
No2 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 1 min	99	97	95
No 5 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 2 min	84	78	70
No6 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 3 min	98	80	60

Harrison further suggested that local differential expansion rates and moisture removal from coal through steam generation may be possible breakage mechanisms insofar as chemical analyses (Table 5.6) had shown considerable reductions in total moisture content following microwave treatment.

Table 5.6 Chemical analysis on samples microwaved at 0.65kW (Harrison 1997)

	C (%)	H (%)	N (%)	Cl (%)	CO_2 (%)	Moisture content (%)	Ash content (%)	Calorific Value (Kj/kg)
No2 coal								
Non-treated	69.1	4.34	0.86	0.51	0.34	5.2	13.5	29040
Exposure 1 min	69.0	4.50	1.54	0.50	0.36	4.4	14.4	28640
Exposure 3 min	69.4	4.65	1.59	0.50	0.41	3.7	13.5	28920
Exposure 5 min	68.9	4.50	1.58	0.50	0.21	2.9	13.9	28740
Exposure 8 min	69.7	4.56	1.56	0.49	0.26	2.1	13.6	28800
No5 coal								
Non-treated	67.7	4.26	1.53	0.18	0.40	1.9	19.0	28400
Exposure 5 min	67.7	4.39	1.57	0.19	0.04	1.5	17.6	28680
No6 coal								
Non-treated	63.8	3.74	1.37	0.16	0.54	3.4	21.1	26780
Exposure 5 min	62.7	4.06	1.33	0.23	0.46	1.9	22.3	26180
Exposure 7 min	68.0	4.37	1.51	0.24	0.38	1.8	16.1	28460

The analyses also showed slight reductions in calorific value as a consequence of microwave exposure and suggested that some volatile gas may also be liberated.

Estimates for the microwave energy consumption were made based on 5 minutes exposure at 0.65kW. Using Equation 5.1, Harrison estimated that approximately 108kWh/t energy would be necessary to produce the improvements in grindability (approximately 70% RWI) from microwave energy. However, the milling energy required to produce pulverised fuel is 15-40kWh/t, depending upon coal rank (Spiers 1952). Hence, a substantial increase in process energy requirements would be needed with the inclusion of a microwave cavity. However, Harrison (1995) compares this energy requirements with that for conventional convective drying methods. Based on muffle furnace heating from ambient temperature to temperatures comparative to 5 minutes exposure to microwave radiation at 0.65kW (290°C), a total of 94kWh/t energy (assuming 100% heat transfer) would be necessary (Equation 5.2). Whilst microwave-assisted is more costly than the use of commercial plant alone, one advantage of the former is the potential for ash and sulphur reduction.

$$Q = Pt \tag{5.1}$$

$$Q = MC_p\theta/t \tag{5.2}$$

Q	Energy absorbed or produced (J/s)
M	Weight of sample (kg)
C_p	Specific heating capacity (kJ/kg)
θ/t	Heating rate (°C/s)

5.7 Effect of conventional drying upon coal grindability

Conventional vertical spindle mills producing pulverised fuel are swept with hot transport air at 80°C. Coal surface moisture is thereby reduced. However at higher temperatures, inherent moisture can also be removed. Lytle et al (1992) found that by subjecting medium volatile coals to convective heating at 204°C, grindability could be improved. Pretreated and non-treated coals were each ground in a steel ball mill for 60 minutes, results show a relative reduction ratio of 1.4 indicating a significant change in comminution behaviour due to preheating.

Harrison (1995) heated three-coal blends in a muffle furnace at temperatures above 200°C to compare their grindability with microwave treated samples (Section 5.6). The coals were ground for 5,20 and 45 minutes in a rod mill and the size distributions compared using the Relative Work Index (Berry 1966).

Table 5.7 The Relative Work Index of sub-bituminous coals after muffle furnace treatment (Harrison 1997)

	Relative Work Index %, ground for:		
No 2 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Heated for 1 hour at 220°C	92	79	80
No5 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Heated for 1 hour at 290°C	90	88	80
No6 coal	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Heated for 1 hour at 226°C	96	82	65

Results (Table 5.7) are reasonably similar for No5 and No6 coals after microwave treatment for 5 and 7 minutes respectively at 0.65kW (Table 5.5). However, microwave treatment further improves the grindability of No2 coal. The results do show that conventional drying improves coal grindability and confirms that moisture could be a major influence on coal comminution behaviour (Harrison 1995).

5.8 Conclusions-Review of the effect of microwave radiation on coal

Research has shown that improvements with coal drying, grindability, desulphurisation potential and pyrolysis can occur from the use of microwave technology. The investigations suggest differential heating and associated differential expansion rates of coal and mineral inclusions combined with steam generation as significant features of all of these processes. Microwave technology has also been applied to on-line moisture determination (Bates 1989) and as an aid to iron reduction using coal as the reducing agent (Zhong 1996).

Previous studies have shown great potential for application of microwave technology within the coal industry i.e. desulphurisation, demineralisation and drying. Despite substantial work reported in these fields, further research is required to quantify the effect of microwave radiation on coal processing. In particular, what effect does microwave radiation have on coals of different rank, under what conditions can higher powers be used and is there an optimum coal size for microwave exposure with regards to both desulphuisation and grindability. There is also a need to verify theories on breakage mechanisms and optimisation of microwave conditions. Most importantly, milling plant simulation and full economic evaluation of these processes are necessary to realise the potential for microwave beneficiation of coal.

CHAPTER SIX

THE EFFECT OF MICROWAVE RADIATION ON COAL PRELIMINARY STUDY

6.1 Introduction to experimental studies

The initial aim of this study is to quantify any changes occurring in the chemical and physical properties of coal when it is subjected to microwave radiation.

Preliminary work was conducted using a range of pre-cleaned coals of varying rank (group 1) and a typical range of sub-bituminous and bituminous coals (100% passing 32mm sieve size) to be used as components in a power station blend (group 2).

Table 6.1 Coal reference table

Group 1		Group 2	
Abbreviation	Colliery name	Abbreviation	Colliery name
F-1	Cynheidre	P-1	National power
F-2	Taff Merthyr	P-2	Rossington
F-3	Cwmbargoed	P-3	Kiverton
F-4	Bentinck An	P-4	Harworth
F-5	Ollerton	P-5	15% ash Daw Mill
F-6	Daw Mill	P-6	ECI 2
F-7	Kellingley	P-7	Welbeck
F-8	Nadins	P-8	Gascoigne Wood

It was necessary to investigate the effect of microwave radiation on a selection of coals to determine the factors which influence any changes in coal grindability, handleability and fuel potential. Group 1 coals have been selected on the basis of their coal rank and low ash content. The relationship between microwave grindability and coal rank (group 1) is discussed in Chapter 7.

Group 2 coals have been selected to determine the effect of microwave exposure on typical power station blends.

6.2 Preliminary study on microwave grindability

This section reports the preliminary investigation into the effects that microwave radiation may have upon the grindability of coal. A series of sub-bituminous coals (typical UK power station feed) have been exposed to microwave radiation at varying times of exposure, microwave power and sample load at an operating frequency of 2.45GHz to investigate whether any grindability improvements can be achieved. The milling capabilities, chemical and physical properties of the coals following their exposure to microwave radiation are compared to non-treated samples to ascertain the potential use of microwave heating as a method of reducing pulverised fuel processing energy requirements.

6.3 Repeatability of proximate analysis

Proximate analyses of group 2 coals have been experimentally determined by the CRE (Coal Research Establishment) Group and by The University of Birmingham (UB) in accordance to BS1016 part 3 (outlined in Appendix A). Results (shown in Appendix B and Table 6.2) show a sample analysis and demonstrates the accuracy of the test procedure. The differences are mainly in the moisture and ash contents. The tests were carried out at different times of year and therefore surface moisture will change due to storage and relative humidity. The ash content may vary from sample to sample due to the heterogeneous nature of the coals. To compensate for sampling errors; the results shown in Table 6.2 and Table 6.3 are average values from at least five analyses runs per coal and measured within the same day (Relative Humidity ranged from 42 to 43%).

Table 6.2 Proximate analyses (as-received basis unless stated) repeatability results of P-3 and P-1 coals

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
CRE P-1	3.7	18.7	29.8	47.8	38.04
UB P-1	3.7	18.2	29.7	48.4	38.03
CRE P-3	4.7	18.3	30.7	48.3	38.80
UB P-3	4.3	19.2	30.3	46.2	39.60

6.4 Proximate analysis of group 2 coals

Group 2 coals differ from those in group 1 in the way they were mined. Group 1 coals have been specially extracted from the middle of the coal seam and as much have low mineral matter impurities. Group 2 coals are 'as-received' coal blends extracted using typical mining methods and subsequently subjected to a preparation process which removes only part of the gangue 'ash' materials.

Table 6.3 Proximate analysis (as-received basis unless stated) of group 2 coals

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
P-1	3.73 (0.15)	18.20 (0.02)	29.69 (0.23)	48.39	38.03
P-2	4.42 (0.09)	13.45 (0.06)	32.25 (0.20)	49.88	39.27
P-3	4.31 (0.09)	19.20 (0.01)	30.29 (0.23)	46.20	39.60
P-4	2.26 (0.17)	7.70 (0.54)	34.45 (0.23)	55.60	38.26
P-5	4.09 (0.06)	13.54 (0.01)	34.08 (0.12)	48.29	41.37
P-6	4.41 (0.17)	12.91 (0.06)	30.33 (0.32)	52.35	36.68
P-7	3.56 (0.08)	6.10 (0.02)	34.02 (0.13)	56.32	37.66
P-8	3.17 (0.05)	21.87 (1.31)	26.72 (0.36)	48.24	35.65

Results (based on the mean value of 5 samples for each coal) of the proximate analyses undertaken on group 2 coals are presented in Table 6.3 and indicate that the coals range in rank from the lowest ranked coal; P-6 coal to the highest P-5 all being sub-bituminous. Standard deviations for the analyses are shown (in brackets) in Table 6.3, all values are within acceptable limits outlined in BS1016.

6.5 Ultimate analysis of group 2 coals

Ultimate analyses in accord with BS1016 (part 6) were determined on group 2 coals. Results (Table 6.4) show that the coals exhibit a similar pattern. In particular, their sulphur and nitrogen contents are low and the carbon, hydrogen and oxygen contents are higher. Ultimate analyses are expressed on a dry mineral matter free basis and only considers the organic coal component.

Table 6.4 Ultimate analysis (dry, mineral matter free basis) of group 2 coals

Coal sample	Sulphur %	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
P-1	1.95	65.70	4.13	1.44	26.19
P-2	1.68	69.10	4.34	0.86	23.17
P-3	1.83	68.30	4.16	1.14	24.02
P-4	2.82	67.70	4.26	1.53	23.11
P-5	2.18	67.70	4.65	1.30	24.02
P-6	1.55	67.70	3.93	1.19	24.34
P-7	-	-	-	-	-
P-8	1.33	84.50	4.90	1.80	6.5

6.6 Ash, forms of sulphur and normative analysis of group 2 coals

The normative (ash) analysis is appropriate to studies of the individual gangue mineral inclusions within the coal structure. Some inclusions may absorb microwave energy and hence have a direct effect upon coal structure. The normative analysis is derived from chemical analyses and assumed molecular balances for associated common minerals. Ash analysis is used to determine the oxidised mineral constituents produced from total combustion of the coal. Typically, ash analysis relates to the mass percentage of the most common eleven inorganic products which result from coal combustion. Group 2 ash analysis is shown in Table 6.5.

Table 6.5 Ash analysis (percentage based on ash content) of group 2 coals

Coal	**P-1**	**P-2**	**P-3**	**P-4**	**P-5**	**P-6**
Na_2O %	1.1	2.2	0.8	0.7	0.4	0.4
K_2O %	3.6	2.9	2.6	3.2	2.0	3.0
CaO %	2.1	2.9	2.6	1.0	7.4	1.8
MgO %	1.5	1.5	1.6	1.2	2.0	0.9
Fe_2O_3 %	10.8	10.4	12.9	16.9	9.3	12.1
Al_2O_3 %	26.7	24.2	24.1	23.5	21.0	26.1
SiO_2 %	51.4	52.7	52.9	51.9	50.9	54.0
SO_2 %	1.3	1.9	1.5	0.2	0.7	1.3
TiO_2 %	0.8	0.8	0.1	0.1	0.2	0.1
Mn_2O_4 %	0.1	0.1	0.1	0.1	0.2	0.1
P_2O_5 %	0.3	0.2	0.5	0.4	0.7	0.3

Clearly, the major inorganic elements are silica, iron, aluminum and calcium. This information-combined with the proximate analysis, ultimate analysis (including chlorine and carbon dioxide) and the forms of sulphur analysis (Table 6.6) help in determining the original mass of each inherent gangue mineral prior to combustion.

Table 6.6 Forms of sulphur (dry basis) of group 2 coals

Coal	**P-1**	**P-2**	**P-3**	**P-4**	**P-5**	**P-6**	**P-7**
SO_4	0.18	0.05	0.12	0.24	0.06	0.40	0.29
Pyritic	0.67	0.52	0.67	1.14	0.52	2.04	0.66
Organic	1.06	0.90	0.91	1.24	1.23	0.97	1.25
Total	1.91	1.47	1.70	2.62	1.81	3.41	2.20

Table 6.6 also shows that all the coals naturally contain varying quantities and forms of sulphur, the sulphur being measured as either sulphate or expressed as organic or pyritic forms. This analysis is required to determine the mineral contents of iron as pyrite; this is relevant to further chapters concerning sulphur emission technology.

Appendix B contains information on the normative analysis results for both group 1 and group 2 coals. The results firstly show that the most common minerals associated with the group 2 coals are mica, kaolin and quartz. Smaller quantities of dolomite, pyrite and ankerite are present. Previous studies have suggested that sulphide minerals such as pyrite are significant receptors of microwave energy whilst the other aforementioned mineral species are poor absorbers. The preliminary implication is that there may be a possible source of ignition, i.e. pyrite, which will heat at a faster rate than the bulk of the coal. Care was taken during microwave experiments and any particles containing hotspots (observed by the distinctive colour and odour of sulphurous gases) were removed immediately.

6.7 Standardization of grindability studies

As the principle objective of this study is to determine whether microwave irradiation can affect coal grindability. A test was developed based on the use of a laboratory scale rod mill in which coal samples (identical apart from the extent of their microwave treatment) were subjected to a standardised milling treatment. The rod mill was chosen because of its availability and ease of use. The mill, of mild steel construction, was of internal dimensions 155mm (diameter) and 280mm (length). Each rod was 270mm (length) and 25mm (diameter); up to 9 rods could be used. Mill operating conditions were selected from a preliminary evaluation of coal breakage behaviour before and after microwave treatment. Grindability indices were based on size distribution analyses (made in accordance with BS410) of the feed and milled coal. The resulting size distribution data was then expressed in terms of a Relative Work Index (Chapter 4). However, it was accepted that standard grindability tests such as the Bond and Hardgrove indices were not appropriate due to their sample preparation requirements (required maximum particle size and sample volume) and milling time.

6.8 Selection of mill operating conditions for grindability studies

6.8.1 Quantity of coal for batch milling

The charge to the mill must be sufficient to represent a true sub-sample of the whole batch and capable of providing reproducible results after allowing for losses from sampling and handling. Coal charge quantity effects were examined at three levels: 250g, 500g and 1000g. Samples of the same initial size distribution were used. The mill operating features were maintained constant; in particular, the speed of rotation was 100rpm and the mill contained 7 rods. Tests were confined to one coal (P-1) and the coal product size distributions were measured after 5 and 45 minutes of milling.

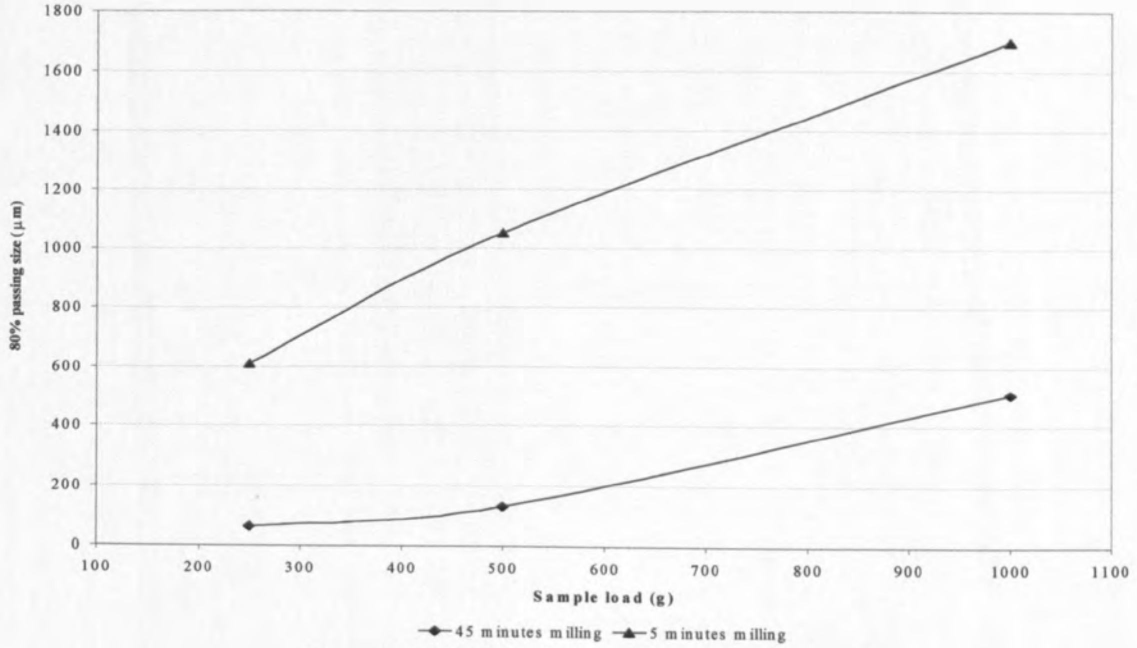


Figure 6.1 Variation in D_{80} with mill load

Results (Figure 6.1) show for a given grinding time, the smaller the coal quantity charged to the mill, the greater the extent of size reduction. After 45 minutes milling, 80% of the 250g and 500g coal charges were below 130μm. In contrast, a 1kg coal charge gave an 80% passing size in excess of 500μm. The implication is that the large particles which are predominantly broken by impact are reduced in size at a higher rate as the coal charge quantity decreases. At lower charge loads there are fewer large particles that require breaking before the major grinding mechanism becomes one of attrition. The results also suggest that size distribution converges to a point where all the particles will be at a specific size, the lower the initial sample load the less milling is required to achieve this point. It is noted that some fine material is lost during sieve analysis. To reduce experimental errors in handling fine particles and in consideration of the milling time necessary to achieve pulverised fuel size (80%<76mm) a 500g coal charge was chosen as the standard quantity.

6.8.2 Number of rods

Cited literature (Wills 1992) states that, for maximum size reduction, the optimum volume occupied by the mill charge (grinding media and material being ground) should be between 40-75% of the internal volume of the mill. For coal quantities of 500g, tests where conducted to determine the relationship between size reduction and the number of rods used in milling. The speed of rotation was 100rpm and the number of rods used were 5, 7 and 9 respectively. Milling times were 5 and 45 minutes.

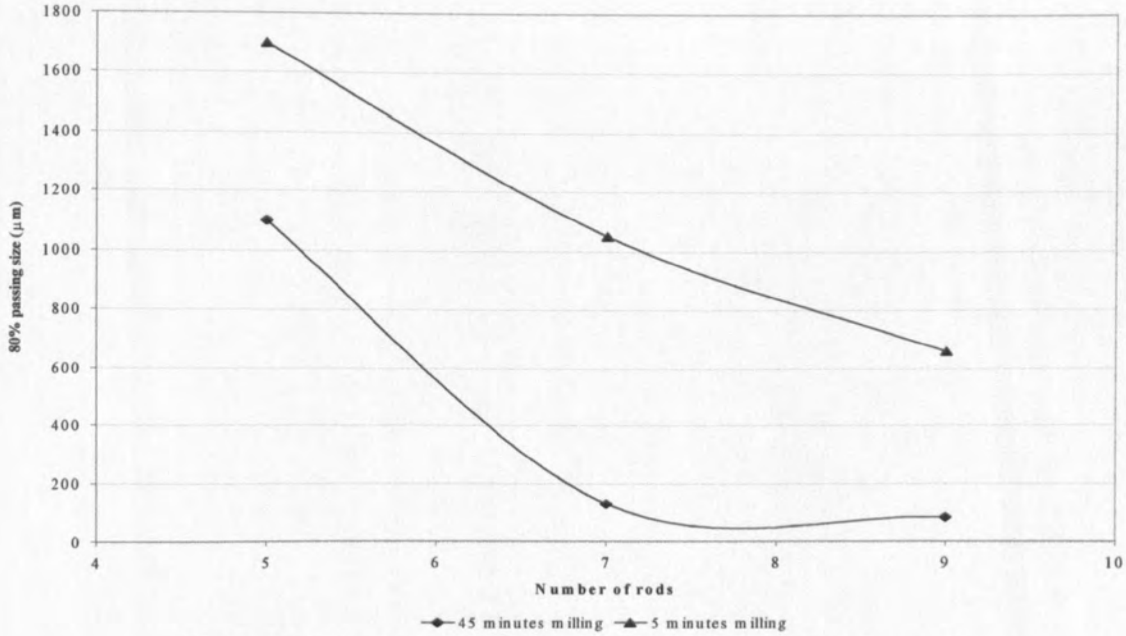


Figure 6.2 Variation in D_{80} with the number of rods used

Figure 6.2 shows that, for 5 minutes milling, as the number of rods are increased, the 80% passing size of the ground product decreases. This indicates a greater effective grinding action. A similar trend is observed for 45 minutes milling (5 and 7 rods) although this is not sustained when the number of rods is increased from 7 to 9 where there was little change in grinding characteristics. Accordingly, as final product size characteristics were achieved using either 7 or 9 rods, the number of rods used in subsequent tests was set at 7.

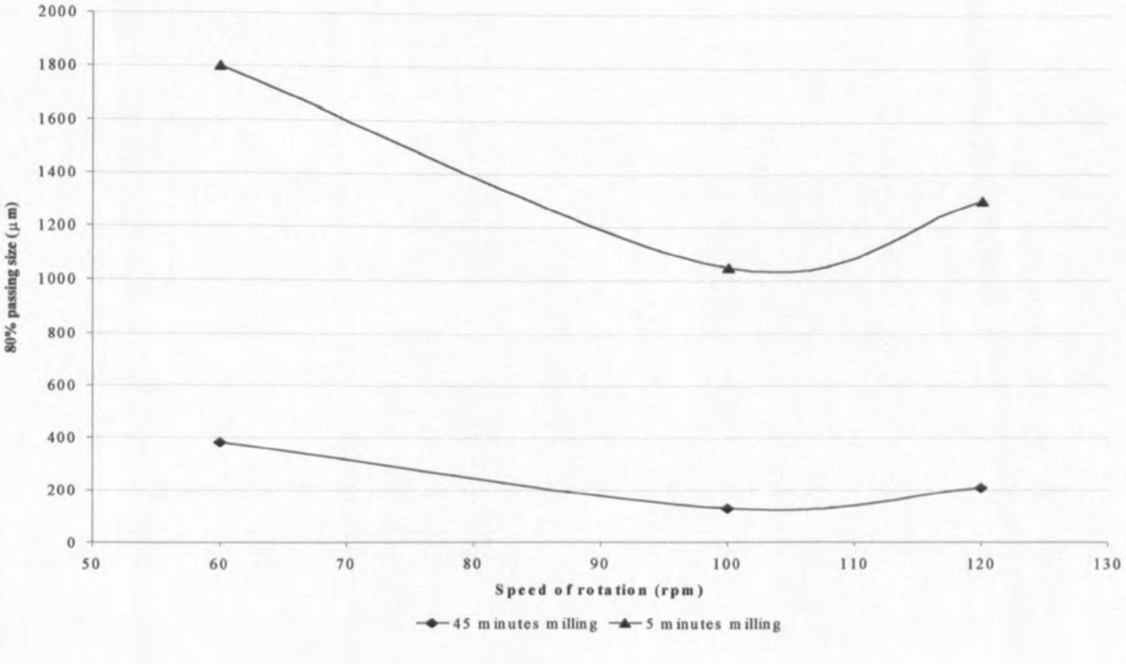
6.8.3 Mill speed

In common with other forms of tumbling mills, breakage during rod milling arises from a combination of impact and attrition. Clearly, the speed of rotation of the mill and number of rods within the mill have major influences on grinding performance. If the speed of rotation is too low, then only a cateracting motion is generated and the relatively mild stresses may have little effect on the breakage of coarser particles. If the speed of rotation is too high the rods would become 'pinned' to the mill wall (centrifugal force) thereby impairing both impact and attrition. The speed at which 'pinning' initially influences the mill performance is defined as the 'critical velocity' and can be evaluated numerically from Equation 6.1.

$$V_c = \frac{42.3}{\sqrt{D-d}} \tag{6.1}$$

V_c	Critical speed (rev/min)
D	Diameter of the mill (m)
d	Diameter of the rod (m)

The critical velocity based upon dimensions of the current mill was 117rpm (Equation 6.1). Test results show that increasing the speed of rotation from 60rpm to 100rpm increases the product fineness. Evidently, breakage by impact and attrition has been enhanced by the increase in mill speed. However, when the mill speed was raised from 100rpm to 120rpm, the product become coarse, although calculations had shown that the critical velocity had not quite been reached (as indicated by the change in size distribution) the cascading effect may have been reduced. This would reduce impact breakage but not attrition. From Figure 6.3 and a consideration of the grinding performance of other group 2 coals; 100rpm was considered as an approximate optimum speed.



6.3 Variation in D_{80} with speed of rotation

6.9 The effect of rod milling on coal

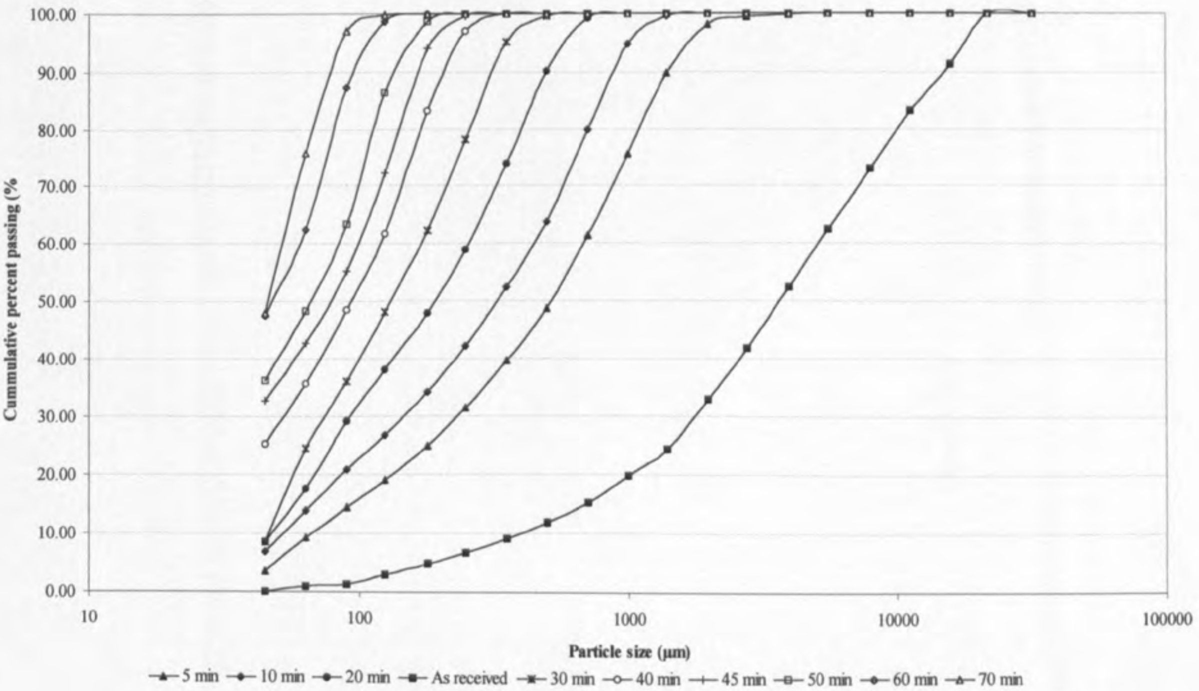


Figure 6.4 Size distribution of P-1 coal with increasing milling time

Two (500g) representative samples of P-1 coal were used to determine if microwave radiation has any effect on coal grindability and to determine the size distribution effects produced by rod milling over time. The original size distribution from a batch sample of approximately 30kg was determined, the two 500g P-1 coal sub-samples were made representative to the original size distribution. Size distribution measurements were made after every 10 minutes of milling. Milling conditions were as described previously (7 rods used at a rotational speed of 100rpm). Figure 6.4 shows the size distributions of an as-received P-1 coal sample with increasing milling time. Figure 6.4 also shows that initially the D_{80} is reduced rapidly over the first five minutes of milling; this is expected due to the selected impact (and hence breakage) of coarse particles. As attrition become the more predominant grinding mechanism, the size distribution converges creating a more closely sized product. This is consistent with theories on the grindability of materials in rod mills. The PF size distribution characteristic used in power generation is 100% passing 125µm with a D_{80} of 75µm. This characteristic is achieved within 60-70 minutes rod-milling of P-1 coal. The second P-1 coal sample was retained for microwave exposure prior to milling.

6.10 The effect of microwave radiation exposure on coal grindability

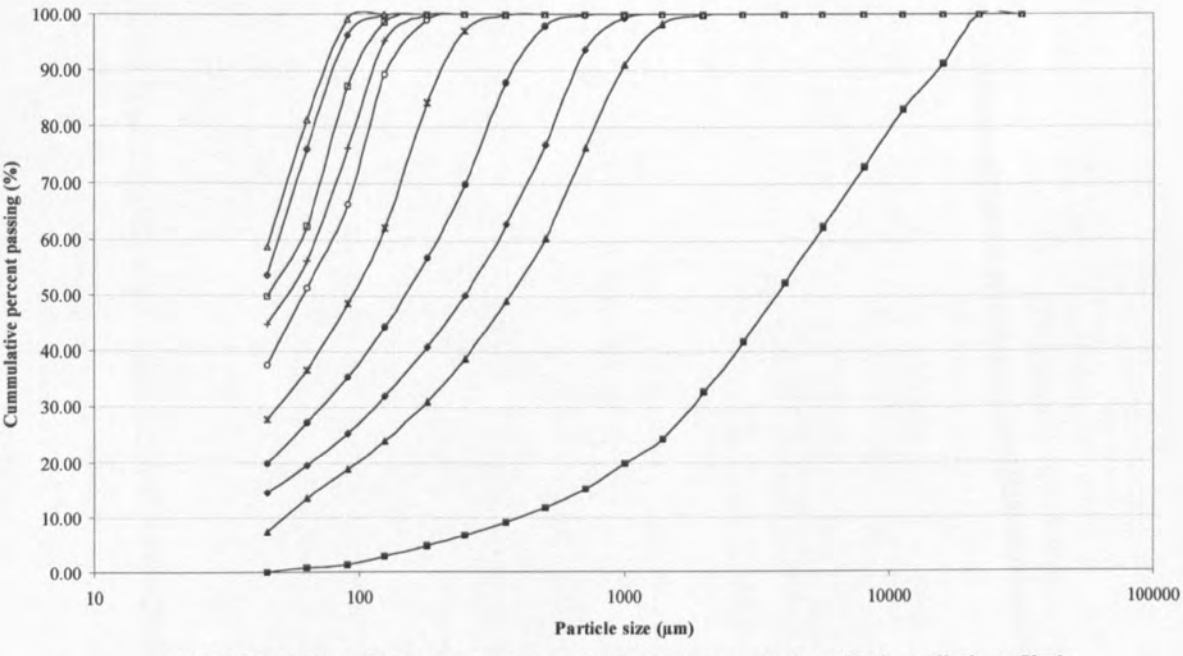


Figure 6.5 Size distribution of P-1 coal with increasing milling time following microwave exposure at a power of 0.65kW and at a frequency of 2.45GHZ for 8 minutes

A 500g representative coal sample (P-1) was exposed to microwave radiation at a power of 0.65kW and frequency of 2.45GHz for 8 minutes. The sample was then milled for 70 minutes (test conditions as described in Section 6.8), with the product size analysis being determined at intervals of 10 minutes. The data (Figure 6.5) shows a similar product size convergence as noted previously (Figure 6.4). However, a D_{80} of 800μm was achieved (with microwave pretreatment) as compared to 1050μm for a untreated sample after five minutes of milling (Figure 6.4). A product of size characteristic is equivalent to that of PF size was attained after 50-60 minutes milling (microwaved sample) compared with 60-70 minutes for the un-treated sample. Clearly, microwave treatment has had a beneficial effect on coal grindability; in particular, the coarser particles are the most affected. Following rod milling for 45 minutes, the Relative Work Index (RWI) (Equation 4.9) of the P-1 coal compared with the untreated coal was 85.4%.

$$\mathrm{RWI} = \frac{\dfrac{10}{\sqrt{Mn}} - \dfrac{10}{\sqrt{Fn}}}{\dfrac{10}{\sqrt{Mt}} - \dfrac{10}{\sqrt{Ft}}} \times 100 \qquad (4.9)$$

RWI	Relative Work Index (%)
Fn	80% passing size of the feed coal, non-treated coal (μm)
Mn	80% passing size of the milled coal, non-treated coal (μm)
Ft	80% passing size of the feed coal, microwave treated coal (μm)
Mt	80% passing size of the milled coal, microwave treated coal (μm)

6.11 The effect of microwave exposure time on coal grindability

Representative samples were taken from 30kg batches of P-2, P-3 and P-5 coals using conventional cone, quartering and riffling techniques. Each sample was of approximatly 500g weight and of similar size distribution. One sample of each coal was milled for 'as-received' reference purposes and subsequent samples were given 1,3,5 and 8 minutes exposure time in a microwave cavity operating at 0.65kW power input and a frequency of

2.45GHz. The average temperatures within the bulk of the coal were recorded immediately following exposure and measured using a standard thermocouple (K-type). Size distributions were determined after 5,20 and 45 minutes milling (test conditions described in Section 6.8) and the results were expressed in the form of RWI (Table 6.7).

Table 6.7 The effect of microwave exposure time on RWI of sub-bituminous coals (0.65kW, 2.45GHz)

	Relative Work Index %, ground for:		
P-5	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 1 min	99	94	94
Exposure 3 min	88	92	94
Exposure 5 min	-	-	-
Exposure 8 min	82	90	92
P-3	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 1 min	100	100	100
Exposure 3 min	58	80	80
Exposure 5 min	56	72	49
Exposure 8 min	55	70	49
P-2	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
Exposure 1 min	85	93	92
Exposure 3 min	80	85	89
Exposure 5 min	77	89	90
Exposure 8 min	71	-	43

Table 6.7 indicates that some coals are more sensitive to microwave radiation than others. In particular, after 8 minutes exposure to radiation, the RWI of P-5, P-3 and P-2 are all different after 45 minutes milling. It is expected that coal rank may have an influence on the receptiveness to microwave radiation. However, these coals were of similar rank, although there were differences in their feed size distributions. Accordingly, it is difficult

to draw detailed comparisons between coals. This specific issue is addressed in later sections (Chapter 7).

Results presented in Table 6.7 indicate that the RWI after 5 minutes milling are significantly lower than those determined after 45 minutes of milling. Accordingly, it is possible that microwave treatment has more of an influence on impactive modes of grinding and hence maybe of greater significance for use on coarser particles. Rod milling generally result in a narrow product size distribution and this convergence may be responsible for the increase in RWI between 5 and 45 minutes milling.

Results (Table 6.7) show that after a microwave exposure period of 1 minute, there is relatively little change in RWI and hence little improvement in grindability. After 3 minutes, the RWI shows a more positive reduction, indicating an improvement in grindability which is broadly sustained for longer grinding periods. Each coal behaves differently. Whilst P-5 shows little change in grindability for microwave exposure periods of up to 5 minutes, the RWI of P-2 coal exhibits a continuous reduction with increasing exposure times and P-3 indicates that grindability maybe at an optimum between 3 and 5 minutes microwave exposure time. The 80% passing size (D_{80}) of feed coals (P-5, P-3 and P-2) introduced into the mill are 10500μm, 13000μm and 16000μm respectively. It appears that the microwave exposure initially has no or little effect, after a period of time an effect upon grindability becomes evident that increases with exposure time. However, this improvement reaches a maximum and any further exposure has no effect on the grindability, this trend appears to depend upon the initial size distribution of the sample i.e. the coarser the feed the longer the exposure which is required and the greater the improvement in grindability.

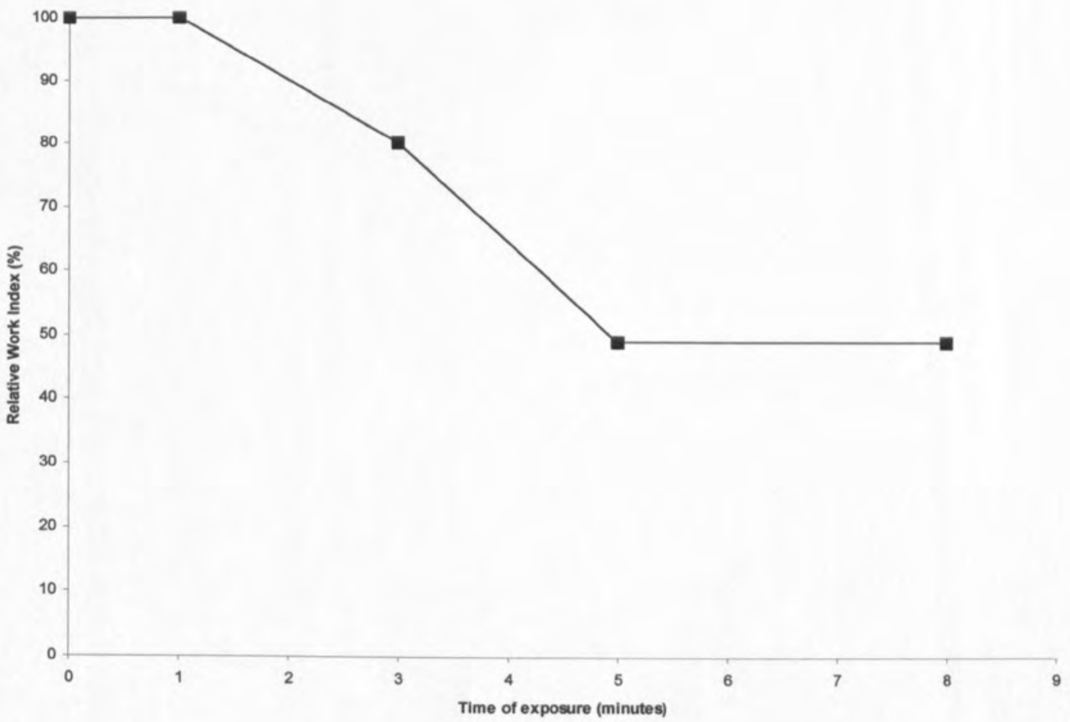


Figure 6.6 Relative Work Index reduction with increased exposure for P-3 coal

To assess the effect of drying on coal grindability, samples of P-2 and P-3 coals were heated in a conventional drying oven prior to milling. The final temperature in the oven was controlled such that it was comparable to that reached by the coals (200°C and 250°C for P-2 and P-3 respectively) following 8 minutes exposure at 0.65kW power (Figure 6.11). Table 6.8 indicates the RWI of the two coals following conventional heating for 3 hours at constant temperature with increasing milling times. Significant reductions were achieved, indicating an improvement to coal grindability. However, the RWI after 45 minutes of milling was not reduced to the same extent of that for samples exposed to microwave radiation for 8 minutes. This feature suggests that microwave treatment can further improve coal grindability over conventional heating.

Table 6.8 The Relative Work Index of sub-bituminous coals with conventional heating after 3 hours

	Relative Work Index %, ground for:		
P-2	**(5 min)**	**(20 min)**	**(45 min)**
Furnace 200°C	77	71	54
P-3	**(5 min)**	**(20 min)**	**(45 min)**
Furnace 250°C	65	92	81

It is suggested that coal sampling techniques used may effect the validity of the results, it is understood that this is due to the sensitivity of the microwave treatment and the differences in feed size distribution. The experiment was repeated using P-2 coal, the initial size distributions of each individual sample remained identical to that of the reference sample. The size distribution of a ~30kg batch was evaluated using a √2 sieve progression series with a top size of 22mm. The series of 500g representative sub-samples were prepared from the retained particles of each size class.

Table 6.9 The Relative Work Index of P-2 coals with increasing microwave exposures (same initial size distribution)

P-2 coal	**Relative Work Index %, ground for:**		
Exposure time	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
3 minutes	90	96	82
5 minutes	77	90	75
8 minutes	80	81	69
10minutes	72	81	63
12 minutes	63	68	54
15 minutes	64	69	54

Samples of P-2 coal were exposed (at a input power of 0.65kW) for 3, 5, 8, 10, 12 and 15 minutes. Results (Table 6.9) show a similar grindability trend as shown previously (Figure 6.6). Following this sample preparation procedure, results show an improved repeatability i.e. two as-received P-2 samples (A and B) were milled for 45 minutes and size distributions taken after 5, 20 and 45 minutes of milling (Figure 6.7).

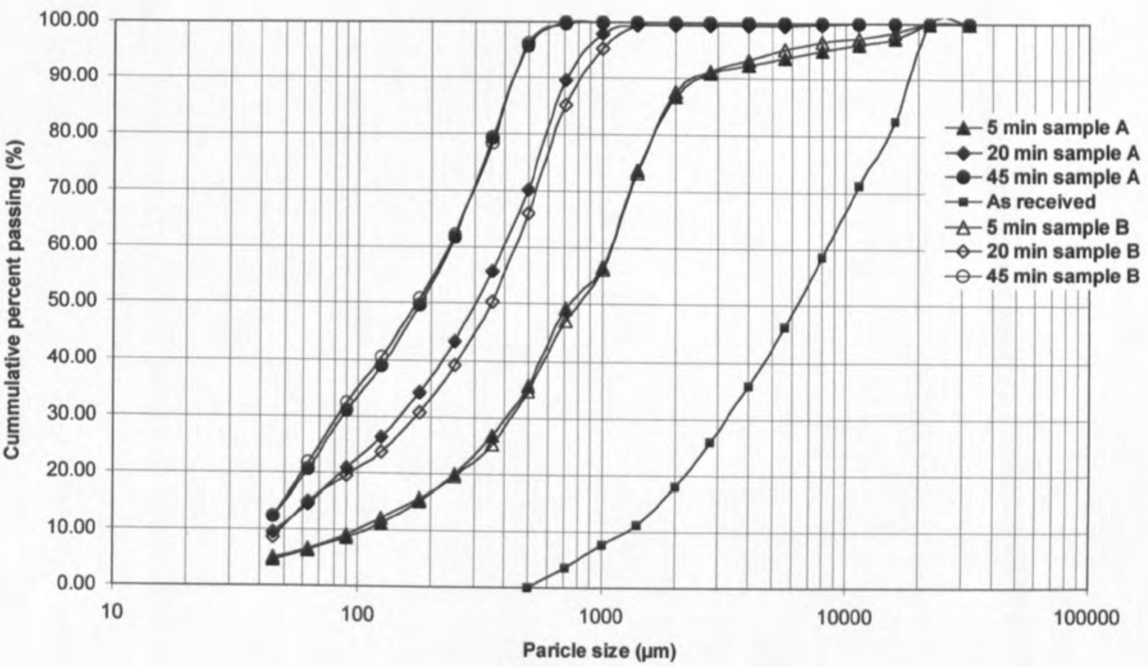


Figure 6.7 As-received P-2 coal rod milled for 45 minutes (two uniform samples, A and B)

6.12 Observed coal structural changes from microwave heating

Fractures have been observed on microwave-exposed coals. Their dimensions and features range from small fractures (10-20μm) detected using Scanning Electron Micrograph imaging (Figure 6.9) to visible cracks (>10mm)(Figure 6.8).

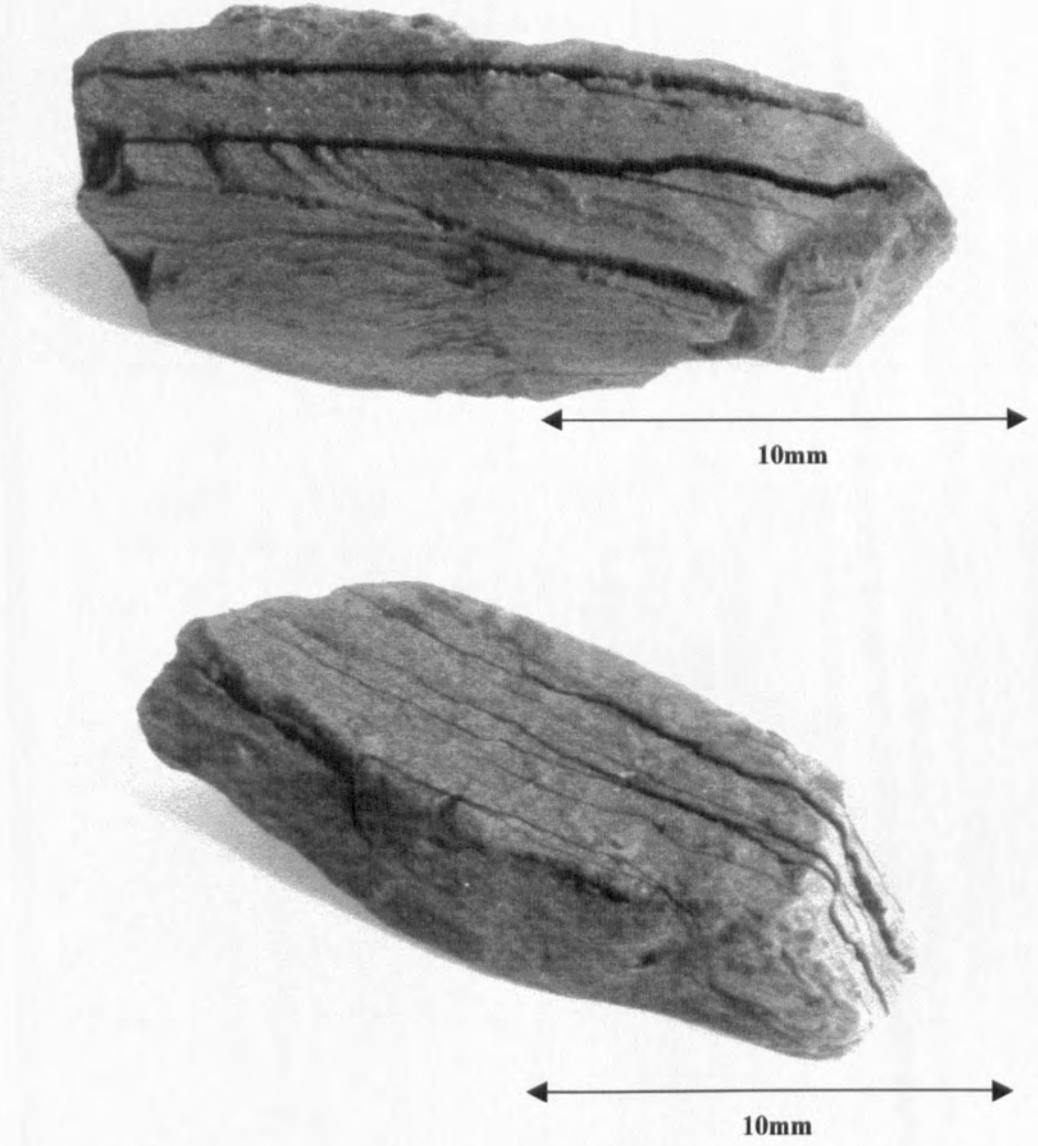


Figure 6.8 Visible fractures produced due to microwave heating (P-8 coal)

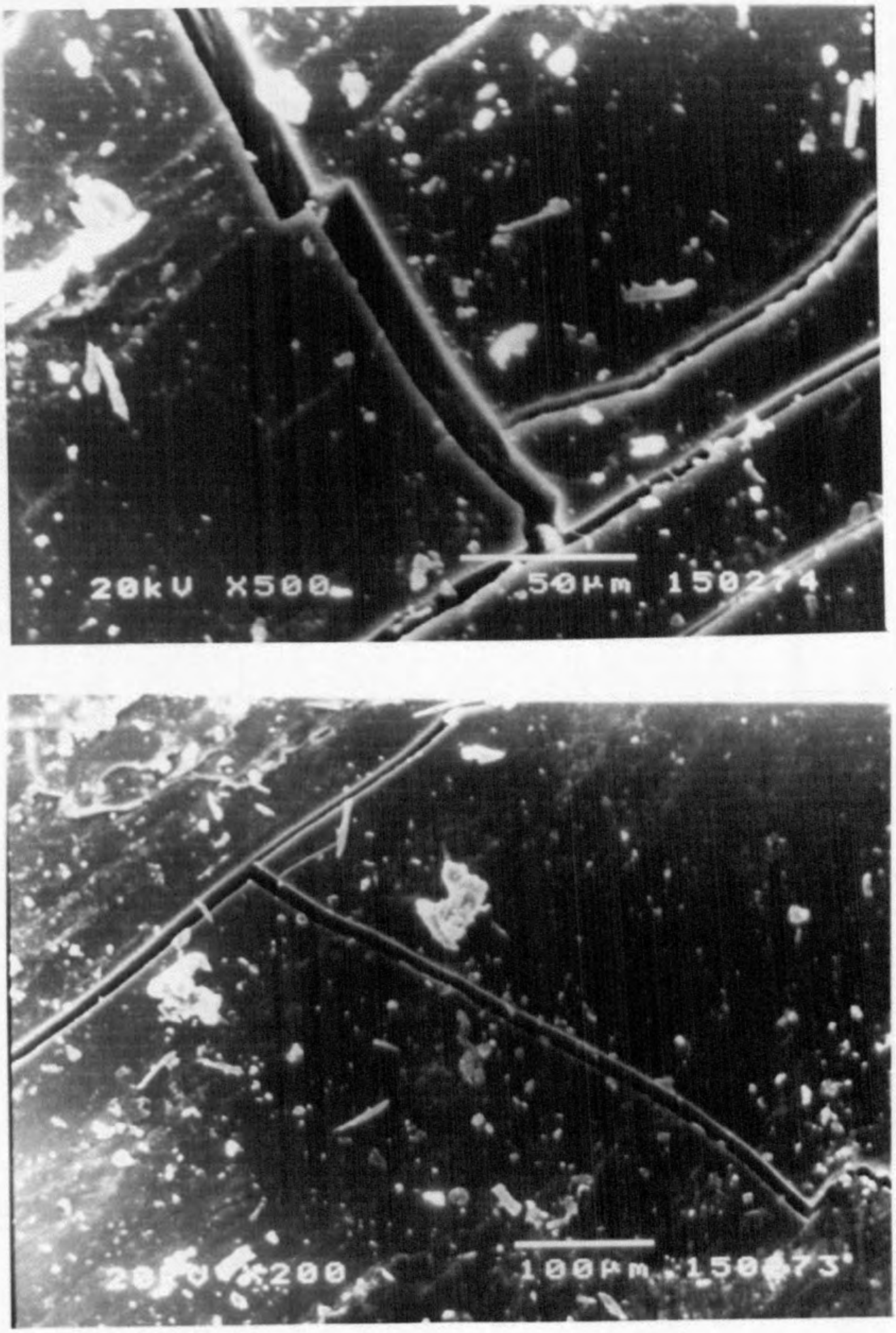


Figure 6.9 Scanning Electron Micrograph image of P-3 microwave exposed coal

The larger cracks were normally associated with the release of sulphurous gas. Previous research has shown that sulphide minerals are rapidly heated within microwave fields (Harrison 1997). Normative analysis and forms of sulphur analysis (Appendix B) have shown the presence of pyrite (FeS_2) within these coals which oxidise during heating. This type of fracture is most probably due to a combination of the release of the sulphurous gases and the rapid expansion of the mineral pyrite. Since there are stringent sulphur limits imposed on coal combustion; most of the coals used in this study contain very small quantities of sulphur bearing minerals (Appendix B). The number of coarse particles observed releasing sulphur was low (Figure 6.8) and accordingly, the number of particles exhibiting these large cracks was also low.

Scanning electron microgaph images (Figure 6.9) indicate that much smaller fractures also occur. Image analysis of microwave treated P-3 coal showed that there small fractures were more common than the larger cracks. Untreated coals exhibited no such fractures (Figure 6.10)

The fractures possibly arise from a combination of gaseous release and stresses produced by the differing expansion rates of gangue minerals within the coal. Pyrite is an extreme case (high heating rate, oxidation and solid volumetric expansion), providing a number of mechanisms explaining the probable causes of the significant improvements in grindability. All minerals heat at different rates within a microwave field and their expansion characteristics differ depending on the local temperature. In some instances-and at certain temperatures-the material changes phase due to the pressures generated by the associated expansion. These changes in the physical size of the minerals and volume occupied by gases promote changes in the structure, weakening the whole particle. It should be noted that cracks may also be initiated upon cooling as this also affects the volumetric changes in the materials and surrounding structure. It is possible that all gangue minerals as well as inherent moisture and (to a lesser extent) the release of some volatile carbon contribute to the production of these smaller fractures. If indeed this is the main mechanism of embrittlement, then microwave grindability would be expected to depend upon the type, size, frequency of appearance and location of gangue minerals as well as specific coal rank.

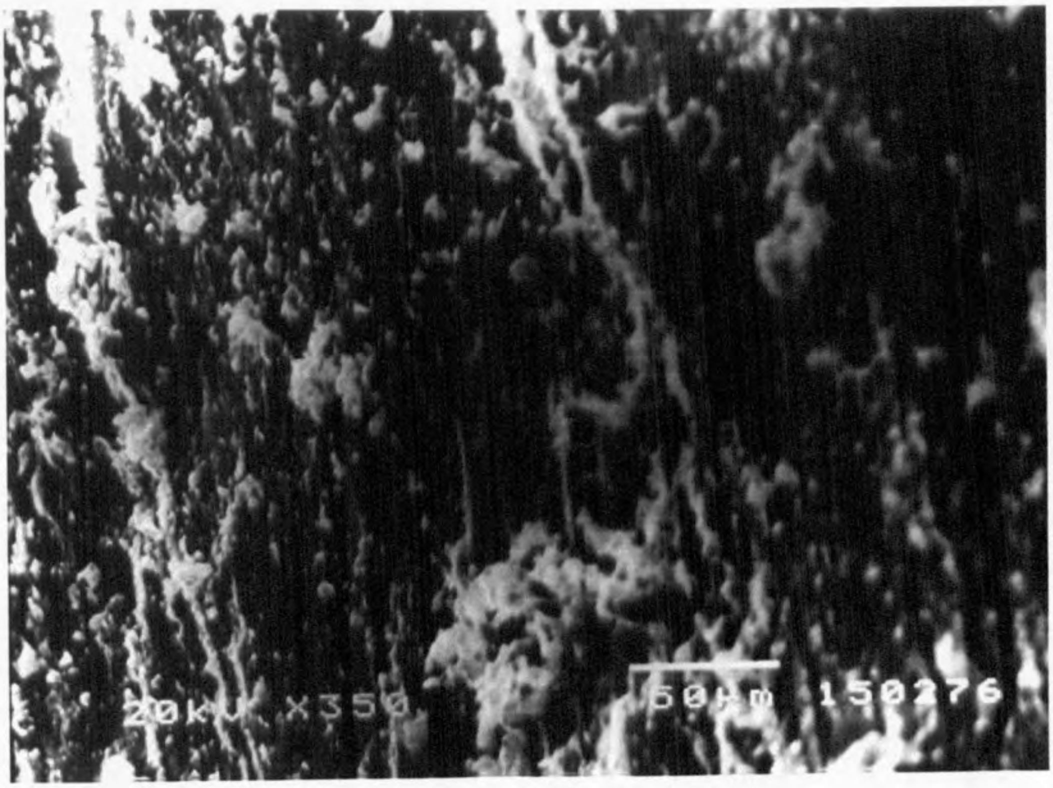


Figure 6.10 Scanning Electron Micrograph image of P-3 untreated coal

6.13 Changes in bulk temperature with microwave exposure

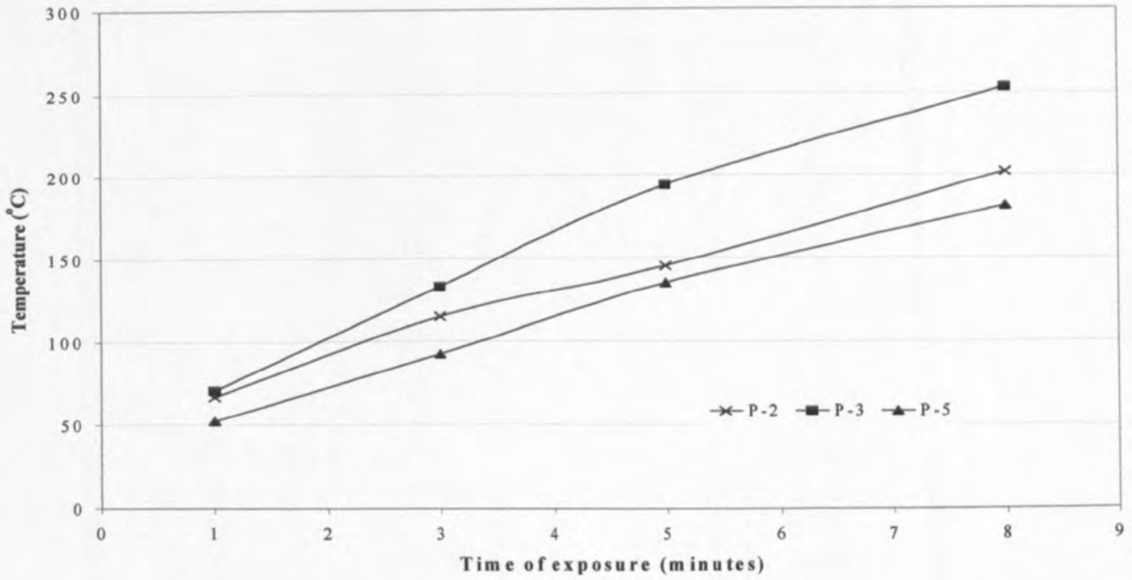


Figure 6.11 Average bulk temperatures of microwave treated coal

The average bulk temperature of the coal was recorded following microwave exposure with the measurements being made using a type K thermocouple (temperature range between –50°C to 950°C). It was shown that after 8 minutes exposure of 500g samples of P-2, P-3 and P-5 coals to a power input of 0.65kW, the final bulk temperatures reached 200°C, 250°C and 175°C respectively. Whilst the maximum bulk temperatures achieved were insufficient to maintain combustion, hotspots were measured in excess of 350°C on some coal surfaces. In particular, minerals (Chapter 8, dielectric properties of minerals and Chapter 11, pyritic oxidation studies) of a high dielectric constant can absorb and heat more rapidly than coal. These hotspots can present an ignition source, and hence care was taken when exposing these coals to microwave radiation. Accordingly, the coals selected for this study did not contain a substantial quantity of iron-bearing minerals which are known to result in high dielectric heating rates.

Figure 6.11 indicates that after 1 minute exposure the bulk temperatures of the three coals remained below 100°C. Consequently, it is expected that insignificant quantities of moisture can be removed from the coal structure. Table 6.7 indicates that little effect on coal grindability is achieved after 1 minute microwave exposure. However, after 3 minutes exposure, the grindability was improved. This suggests that the drying effect of the microwaves may be partially responsible for the improvements in coal grindability and, if this is true, then the total coal moisture content is another variable that must be considered in accounting for the improved grindability of coals following microwave treatment.

6.14 Proximate Analysis of microwave treated coals

A proximate analysis in accordance to British Standards 1016 part 3 was undertaken on P-2, P-3 and P-5 coal samples used in the grindability experiment to determine if microwave radiation has an effect on the basic chemical properties of the coals. The proximate analyses are shown in Appendix D.3. Table 6.10 shows a summary analysis of as-received reference samples and those irradiated for 8 minutes.

Table 6.10 Proximate analysis (as-received basis unless stated) of microwave exposed coal samples

Proximate Analysis	**P-5**	**P-3**	**P-2**
Moisture %	4.09	4.70	4.42
Ash%	13.54	16.30	13.45
Volatile Matter (VM) %	34.08	30.73	32.25
Fixed Carbon (FC) %	48.29	48.31	49.88
$\textbf{Ash}_{\textbf{Dry Basis}}$ **%**	14.12	17.10	14.07
$\textbf{FC}_{\textbf{Dry Basis}}$ **%**	50.35	50.68	52.18
$\textbf{VM}_{\textbf{Dry,mineral free Basis}}$ **%**	41.37	38.86	39.27
Proximate analysis for samples exposed to 0.65kW, 2.45GHz for 8 minutes			
Moisture %	2.55	2.26	2.32
Ash%	10.74	16.08	15.20
Volatile Matter (VM) %	34.61	28.53	30.78
Fixed Carbon (FC) %	52.10	53.13	51.70
$\textbf{Ash}_{\textbf{Dry Basis}}$ **%**	11.02	16.45	15.56
$\textbf{FC}_{\textbf{Dry Basis}}$ **%**	53.47	54.36	52.93
$\textbf{VM}_{\textbf{Dry,miner free Basis}}$ **%**	39.91	34.93	36.53

Table 6.10 shows a significant reduction in moisture content due to microwave exposure, further suggesting the importance of the coal moisture content on microwave grindability. The proximate analysis on a dry ash free basis indicates that there is a small reduction in volatile matter content. Volatile carbon may vaporise at lower temperature than that required for combustion, the reduction may decrease the coal calorific value that would

have otherwise been expected to increase due to the drying effect. To determine the consequences of this reduction in volatile matter the calorific value of the P-3 samples was measured (Table 6.11).

6.15 Calorific value of microwave exposed coal

The calorific values of the coal samples were measured using a Gallenkamp Automatic Adiabatic Bomb Calorimeter in accord with British Standard 1016 Part 5. The calorific value is determined when a known mass of coal is burned in oxygen under pressure in an adiabatic bomb calorimeter. The gross calorific value is calculated from the temperature rise of the water in the calorimeter and the effective heat capacity of the system. A mean of the gross calorific value was calculated from duplicated tests for each coal sample and reported on a dry and ash-free basis.

Table 6.11 Calorific value of P-3 coal (dry, mineral matter free basis) with increasing microwave exposure

Microwave exposure time,	0	1	3	5	8
Calorific Value$_{dmmf}$, (MJ/kg)	36.2	35.2	34.6	34.4	34.1
ΔCV, (MJ/kg)	0	-0.97	-1.59	-1.74	-2.05

Table 6.11 shows that the calorific value decreases as microwave exposure time increases. The total reduction of the calorific value of P-3 coal is approximately 1.75% after 5 minutes of microwave radiation at a power of 0.65kW. Milling studies indicated that the improvement in grindability had maximised for P-3 coal after 5 minutes of microwave exposure and it is therefore accepted that a small reduction in calorific value may result due to the microwave beneficiation.

6.16 The effect of increased microwave power (1.3kW) on coal grindability

Similar grindability experiments were carried out on P-5, P-3 and P-2 coals using an increased microwave power input (1.3kW microwave power instead of 0.65kW). Results show that similar improvements in grindability can be achieved within shorter exposure periods. In accord with results given in Section 6.11, grindability improves with increasing exposure time.

During the tests, it was noted that the samples did not reach an average bulk temperature in excess of 130°C. However, localised hotspots resulted in ignition after exposure times of 2.5 minutes for P-5 and P-2 coals and 3 minutes for P-3 coal. Due to partial combustion, the tests were terminated and it was not possible to demonstrate the full beneficial effect of microwave radiation. The results imply that the use of coals containing materials which are highly absorbent to microwaves is limited by the heating rates of these materials and not the heating rate of the coal. Pyrite may have been the source of ignition, reaching elevated temperatures after a relatively short time. Mineral properties are discussed in subsequent chapters (Chapter 8). However, it is noted that the heating rates of the mineral components do not increase linearly with increasing microwave power input. Clearly, the above experiment indicates the problems associated with the use of increased microwave powers. In particular, ignition may be prevented with the use of inert gas atmospheres but the loss of volatile matter may not be overcome.

The experiments were carried out without prior information of the importance of initial size distribution and hence serves only as an introduction to the problems and advantages of using increased microwave powers. Furthermore, results shown in Table 6.12 are not comparable with those for reduced powers (Table 6.7) as the two sets of experiments were carried out in separate microwave cavities (each of differing volumes and electric field strength distributions) which may affect microwave absorption and hence efficiency (Chapter 8).

Table 6.12 The Relative Work indices of sub-bituminous coals with increasing microwave exposure times (1.3kW, 2.45GHz)

	Relative Work Index %, ground for:		
P-5	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
1 min	96	92	100
2 min	87	98	93
2.5 min	81	74	74
P-3	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
1 min	74	100	100
2 min	72	100	95
3 min	67	92	82
P-2	**(5 min)**	**(20 min)**	**(45 min)**
Non-treated	100	100	100
1 min	94	100	100
2 min	75	75	79
2.5 min	75	81	71

The major disadvantages to increasing the power are attributed to the localised high heating rates of the gangue impurities and the associated transfer of heat to the local coal surfaces which may result in premature ignition. Clearly, partial coal combustion is undesirable as it results in a reduction in fuel potential and uncontrollable emission levels. A positive feature of these results is the reduction in exposure time required to improve coal grindability for the same energy and coal charge; process time may also be reduced as a consequence of increasing microwave power which would favour a continuous process.

6.17 Coal grindability at increased input powers for a given energy input per unit weight

Three samples of P-2 coal were exposed at increasing input power levels from 0.65kW, 1.3kW and 2.6kW for 609s, 305s and 152s respectively, equivalent to 220kWh/t. A variable power input microwave cavity was used (no change in cavity shape and dimensions) and the input energy was selected such that the maximum benefits from grindability tests could be achieved (Section 6.12). The RWI was calculated from the product size distributions following rod milling times of 5, 20 and 45 minutes. Results (Table 6.13) showed that the RWI was broadly independent of power input level providing the energy per unit weight was maintained constant.

Table 6.13 Relative Work indices of P-2 coal with increased power input for a given energy input per unit weight (220kWh/t, 2.45GHz, 500g)

Coal	Power input (kW)	Relative Work Index %, ground for:		
		(5 min)	(20 min)	(45 min)
Non-treated	-	100	100	100
Exposure 609s	0.65kW	69	66	54
Exposure 305s	1.3kW	65	73	56
Exposure 152s	2.6kW	70	70	55

6.18 The effect of batch sample size on power absorption

During preliminary investigations, no attempt was made to optimise the quantity of the coal within the microwave cavity. The tests detailed below show that the microwave cavity in current use (dimensions: length 340mm, width 330mm and height 330mm) can accommodate larger volumes of coal than previously used without affecting coal grindability efficiency.

In particular, batch loads of 250g, 500g and 1kg P-1 coal were used for this study. The microwave power input was 0.65kW. Product size distributions were determined for each load after 45 minutes milling, the 80% passing size characteristics are shown in Figure 6.12.

Duplicate loads were exposed to microwave radiation for 4,8 and 16 minutes respectively so that each sample was irradiated with the same energy per unit weight of coal (173kWh/t). The 80% size characteristics of these (microwave-treated) samples are shown in Figure 6.12. Results indicate that grindability efficiency increases with sample volume.

It is noted that the Relative Work indices are dependent upon mill load (mass of coal and mill medium). The test procedure for RWI calculation was standardised and correlations were made for the 500g samples only (mill load optimum capacity). To calculate RWI for microwave exposed samples of 250g and 1kg a duplicate set of samples were exposed for 4 and 16 minutes respectively (173kWh/t). Two independent 250g samples were treated

(4 minutes at 0.65kW) before milling as a 500g batch. Similarly, the 1kg sample was evenly split into two 500g samples for milling. Figure 6.10 shows the 80% passing sizes for these (500g) milled samples. It can be seen that product sizes (and corresponding RWI) are very similar.

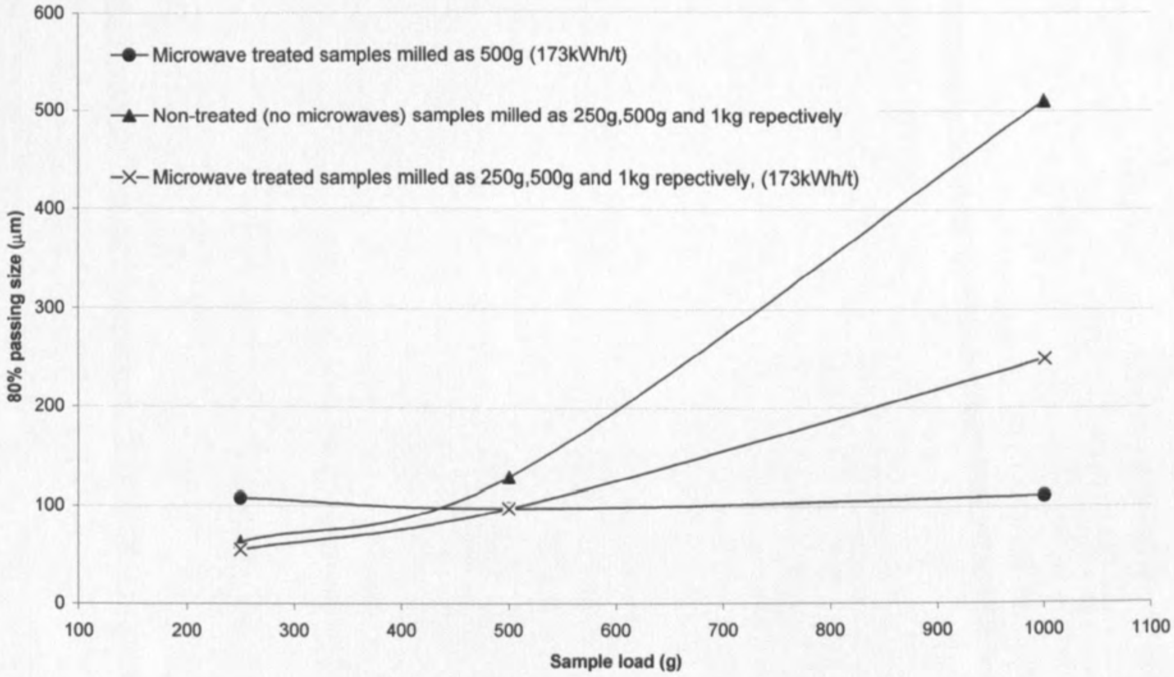


Figure 6.12 Effect of sample load at constant applied microwave energy (173kWh/t)

There is little change to Relative Work Index as sample load increases. However, as sample load increases, grinding efficiency also increases with the range of volumes tested.

6.19 The influence of particle size to improved grindability by microwave radiation

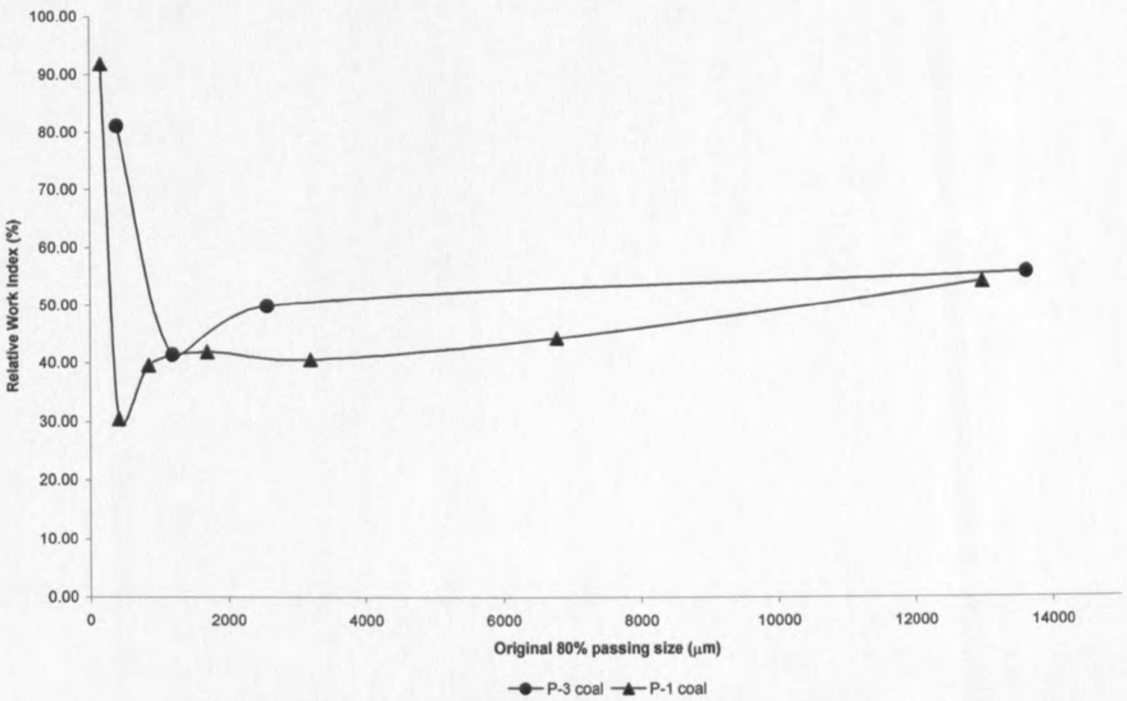


Figure 6.13 Microwave grindability dependence upon initial particle size

Previous experimental work (Section 6.11) has drawn attention to the importance of initial coal size distribution on microwave grindability. It is well known that as particle size decreases, the energy requirements for size reduction increases. It is possible that larger particles gain many small fractures after microwave exposure, whilst smaller particles may produce fewer flaws depending upon the quantity, position, size and distribution of gangue minerals and inherent moisture. In contrast, finer particles are inherently stronger and there is only a low probability of finding a crack.

An experiment was designed to determine the effect of initial particle size on microwave grindability. Samples of P-1 and P-3 coals were separated into size fractions. For each coal size fraction, two samples (500g) were prepared for grindability evaluation. Samples were milled using a rod mill as described previously (Section 6.12) and operated under constant conditions. The final D80's product sizes are given in Table 6.14.

Table 6.14 Influence of particle size on microwave enhanced grindability, 80% product passing size

Sample and particle size range (mm)	Reference product size D_{80} (μm)	Microwave product size D_{80} (μm)
P-1 x>16mm	405	120
P-1 16mm>x>8mm	245	80
P-1 8mm>x>4mm	315	78
P-1 4mm>x>2mm	355	90
P-1 2mm>x>1mm	300	92
P-1 1mm>x>0.5mm	250	87
P-1 0.5mm>x>0.25mm	215	80
P-1 x<0.25mm	73	69
P-3 16mm>x>8mm	272	94
P-3 2mm>x>1.4mm	290	104
P-3 1.4mm>x>0.71mm	310	108
P-3 0.5mm>x>0.09mm	86	68

After converting the product size distributions into Relative Work indices for both coals similar reductions in RWI were shown irrespective of particle size. Inspection of data indicated that for both coals, the fines fractions (X<1mm) have had a slight improvement in grindability after microwave radiation. However, particles greater than 1mm size exhibit relatively large improvements in grindability. Figure 6.13 shows the changes in Relative Work indices with the initial D_{80} particle size. In particular, results show RWI values are between 40%-50% for size fractions greater than 1mm before milling and between 80%-90% for size fractions less than 1mm before milling. Even though all coal particles are affected by microwave radiation, the above results suggest that coal particle size of above 1mm may be most suitable for microwave-induced grindability. It is noted however, that this characteristic may be specific to rod milling.

6.20 Preliminary study conclusions

Laboratory experiments have shown that substantial reductions in Relative Work Index (RWI) can be achieved by microwave exposure of coals. There is evidence to suggest that gaseous evolution (water and volatile matter) as well as gangue mineral expansion are the probable causes for the improvement in coal grindability. The properties of the various coals treated by microwave radiation remained relatively unaltered. No significant change in proximate, ultimate analyses or calorific value were reported. Initial laboratory-scale microwave trials proved successful in weakening the structure of various coal types. However, fine particles (<1mm) were less susceptible to enhanced microwave grindability. Bulk temperatures of up to 250°C could be reached as a result of microwave treatment and localised hotspots may be responsible for some losses in volatile matter content. During preliminary test work no samples were exposed to conditions such as those for coal ignition or combustion.

CHAPTER SEVEN

DEPENDENCE OF COAL RANK RESPONSE TO MICROWAVE TREATMENT

7.1 Introduction-Dependence of coal rank response to microwave treatment

Analyses of results from preliminary studies (Chapter 6) suggest that a combination of factors influence the effectiveness of microwave pretreatment on coal grindability. In particular gas release (in the form of steam, volatile matter and sulphur) and differential expansion of occluded minerals may be controlling features in the propagation of fractures within the coal structure. Coals vary in moisture content depending on their rank (inherent moisture) and (independently of rank) their relative humidity (surface moisture). The gangue mineral content and composition also depend upon the geological location and age of the coal.

Group 1 coals have been selected to evaluate the dependence of coal rank on their response to microwave treatment. It is noted that mineral matter content can vary significantly from coal to coal and even within the same coal seam irrespective of coal rank. Some minerals may attribute (through volumetric expansion) to fracture propagation, and for the purpose of this test, low mineral content coal is preferred. The mineral matter can be minimised by selecting coal samples cut from mid-seam. Group 1 coals refer to such samples (Chapter 6.1) which embrace a wide range of ranks and can therefore differ in carbon structure, inherent moisture content and grindability. It is noted that all coals have been handled in a similar manner and exposed to the same atmospheric conditions before sampling to minimise any possible variations due to relative humidity.

7.2 Proximate analysis of group 1 coals

Group 1 coals represent a suite of individual mid-seam coals, which collectively, cover a wide rank range (lignite F-8 to anthracite F-1). This suite was used to assess the effectiveness of microwave irradiation on coals of different rank and to identify the rank properties of importance. Proximate analyses (BS1016 part 3) indicated that the ash contents of the eight individual coals were below 5% and illustrate the variations in volatile matter, moisture and fixed carbon contents with rank (Figure 7.1).

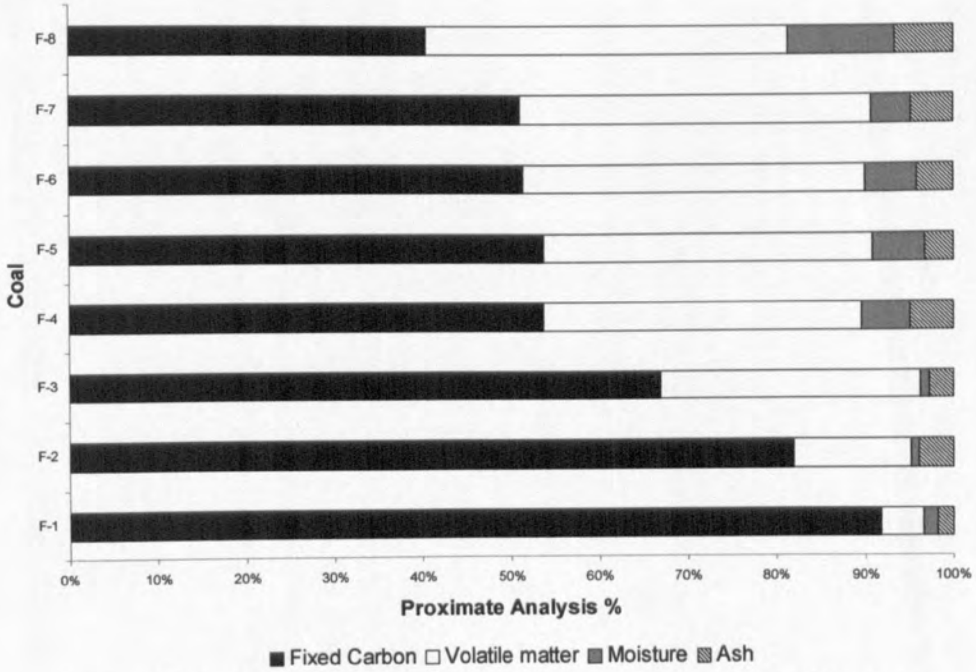


Figure 7.1 Proximate analysis of F-1 to F-8 coals (CRE Group)

Proximate analyses of group 1 coals were determined at CRE Group (Coal Research Establishment) and The University of Birmingham. Results are shown in Appendix B.

Table 7.1 gives data for two coals of widely different rank and demonstrates the inter-laboratory agreement between results. Analysis indicates that the high-rank coal is most homogenous with much less compatible results produced between CRE Group and the University of Birmingham (UB) for the low-rank coal, particularly with the moisture content. The analyses show comparable results irrespective of rank on a dry, mineral matter free basis (Figure 7.1).

Table 7.1 Proximate analyses (as-received basis unless stated) repeatability results for F-1 and F-8 coals

Coal	Moisture %	Ash %	Volatile Matter %	Volatile Matter$_{dmmf}$ %	Fixed Carbon %
CRE F-1	1.6	1.8	4.6	4.8	92
UB F-1	1.5	1.8	6.8	7.0	90
CRE F-8	13.3	7.4	34.9	44.0	44.4
UB F-8	10.8	8.2	35.5	43.8	45.4

7.3 Coal rank and group 1 coals

Table 7.2 Classification of F-1 to F-8 coals

	ASTM classification	**NCB classification**
F-1	Anthracite	Anthracite
F-2	Semianthracite	Bituminous
F-3	Low-volatile bituminous	Bituminous
F-4	Medium-volatile bituminous	Bituminous
F-5	High-volatile A bituminous	Sub-bituminous
F-6	High-volatile A bituminous	Sub-bituminous
F-7	High-volatile B bituminous	Sub-bituminous
F-8	Sub-bituminous B	Lignite

Figure 7.1 shows the proximate analyses as measured by CRE Group. The coal rank can be expressed by correlating the data shown in Figure 7.1 (dry, mineral free basis) with the

information given in Figure 3.2 (Chapter 3). Table 7.2 compares the rank of group 1 coals based on the ASTM system with the coal classification system used by the 'National Coal Board'(NCB) (CRE coal bank 1995).

7.4 Relationship between Hardgrove Grindability Index and coal rank

Properties of coals can be correlated with coal rank, in particular, the Hardgrove Grindability Index (HGI) (a measure of the grinding propensity of coal) is often considered to determine the rank of coals and to compare grindabilities between individual coals (Chapter 3.4).

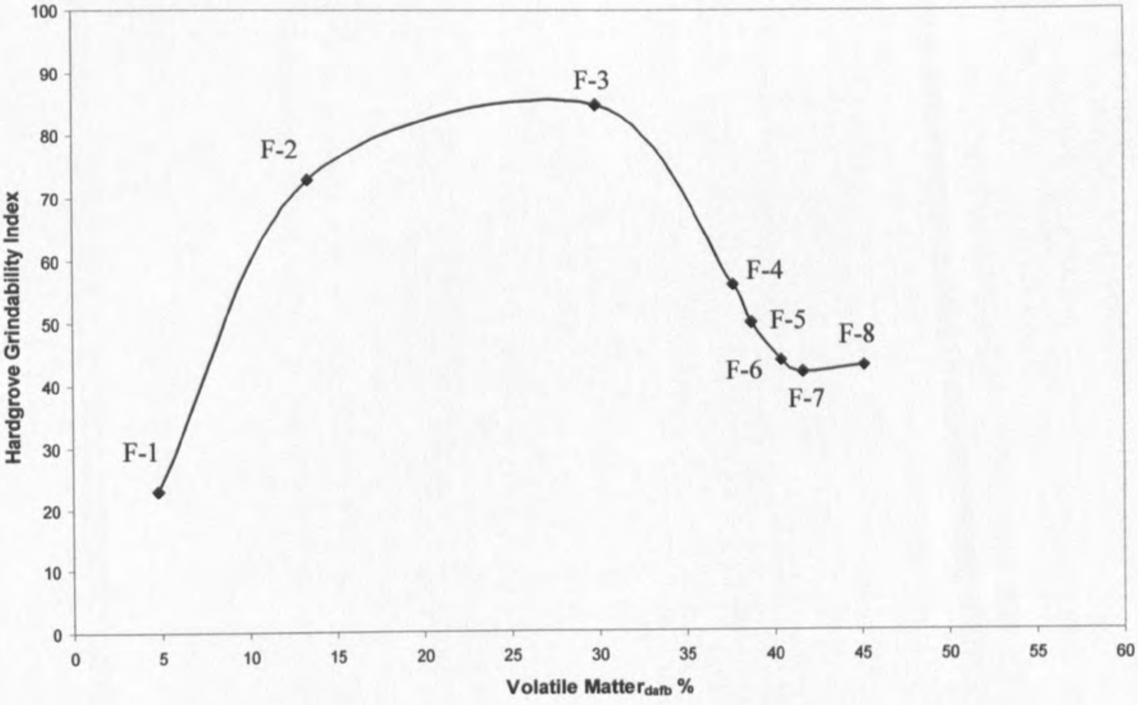


Figure 7.2 The Hardgrove Grindability Index of group 1 coals

The Hardgrove Grindability indices of group 1 coals have been measured by CRE Group in accordance with BS1016 part 112. Results (Figure 7.2) are of the characteristic form

(Chapter 3.4) indicating that the high (F-1) and low (F-6 to F-8) rank coals require more energy for milling (to a given product size) than those of intermediate rank.

7.5 Development of grindability test procedure

During milling, the size distribution of the coal feed may have some influence on its subsequent breakage behaviour and hence the resultant size distribution. In the current tests, the coal feed size distribution was maintained constant, irrespective of coal rank and whether or not the coal had been subjected to microwave treatment. To this end, the coals were pre-crushed in a jaw crusher to a top size of 32mm, suitable for further breakage in a rod mill. Jaw crusher feed and product size distributions are shown in Appendix B. Batches (500g) of the latter were produced by sieving the eight coals (F-1 to F-8), the coals were exposed to ambient atmosphere for twenty four hours before being sealed within air-tight containers to retain their constant relative humidity equilibrium.

The ensuing microwave treatment was constant for all coals, each being irradiated for 8 minutes at 0.65kW power input and at 2.45GHz frequency. This treatment was chosen as earlier experiments (Chapter 6.11) had shown it gave significant benefits for a medium rank coal. Previous and current results could also be compared (Harrison 1995)

In the current tests, milling conditions were identical to those used previously (Chapter 6.8) with the exception that the coal batch weight was 450g. Following microwave treatment, representative sub-samples were taken from each treated coal batch and used for proximate and ultimate analysis, the quantity taken depending upon that remaining after microwave treatment. These samples were placed in vacuum-sealed bags (to prevent changes in total moisture and allowed to cool to room temperature).The coal size distributions at 5, 20 and 45 minutes of milling were determined for both the 450g samples of microwave treated samples and the reference samples in order to determine the Relative Work Index (Chapter 4.2.4) for each coal.

7.6 The effectiveness of microwave radiation on the grindability of coals of different rank

Results (Table 7.3) show similar trends to those illustrated in Chapter 6 (section 6.11) following milling times of 5, 20 and 45 minutes. The results also suggest that microwave radiation has had little effect on the high rank coals (F-1, F-2 and F-3) after a milling time of 45 minutes. Coals of medium and low ranks appear to be more susceptible to grindability effects caused by microwave radiation.

Table 7.3 Relative Work Index of group 1 coals after microwave exposure at a power of 0.65kW and a frequency of 2.45GHz for 8 minutes

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
F-1	90	98	97
F-2	91	79	99
F-3	98	76	97
F-4	82	73	88
F-5	74	66	61
F-6	72	74	74
F-7	68	69	61
F-8	60	66	52

The general trend is apparent (Figure 7.3) i.e. the lower the coal rank the more sensitive the coal to microwave radiation. A possible explanation is that low rank coals contain increased high quantities of inherent moisture. As suggested previously (Chapter 6), inherent moisture would be expected to heat rapidly within a microwave field generating increased pressures during the liquid-gas phase change. It is assumed that an increase in inherent moisture content would propagate an increased number of flaws (Figure 7.4) which would subsequently weaken the coal structure.

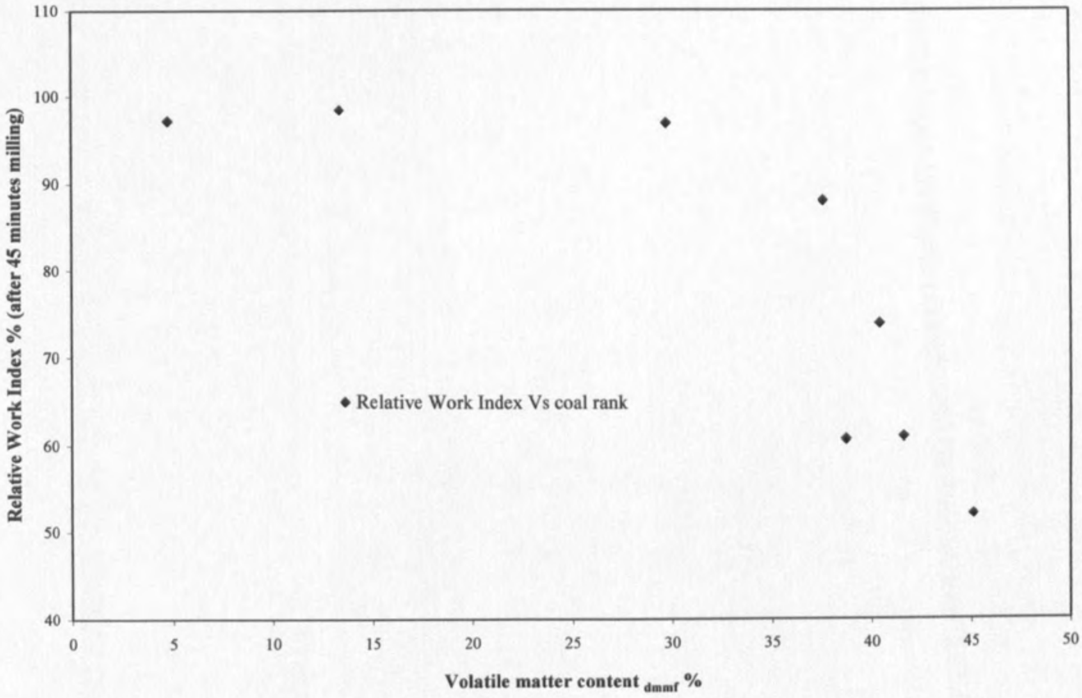
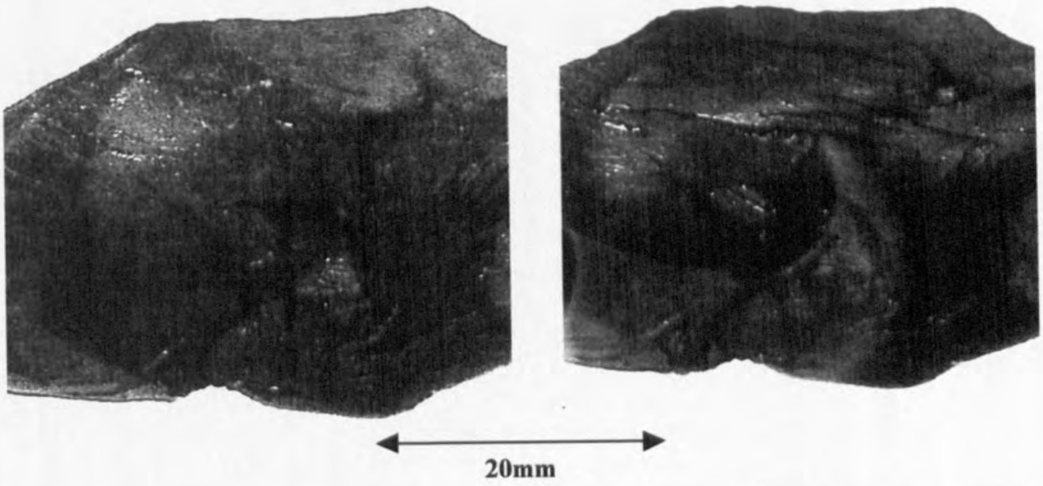


Figure 7.3 Relationship between microwave grindability and coal rank



F-8 coal prior to microwave treatment. **F-8 coal after microwave treatment**

Figure 7.4 Visible fractures after microwave treatment of a low rank coal

7.7 The effectiveness of microwave irradiation on the propensity of coal for fine grinding

The measured Hardgrove Grindability indices of group 1 coals (Figure 7.2) conform to the accepted pattern between grindability and rank in so far as high rank coals (anthracites) and low rank coals (lignites) are shown to be more difficult to grind than those of medium rank. Table 7.4 shows the weight percentage of coal product below 75μm for various milling times for the group 1 coals (Chapter 6.8). Coals were pre-crushed to a limiting top size (32mm) and their individual size distributions show to be identical before milling tests. As expected medium rank coal could be ground finer (Table 7.4) than high and low rank coals. After 45 minutes of milling, medium rank coals (F-2, F-3 and F-4) can produce a typical PF size characteristic (80% passing 75μm). However, as shown in Table 7.4, increased milling time is necessary for the high and low rank coals to produce a similar degree of fineness.

Table 7.4 Degree of fineness after grinding

Coal sample	**Weight percentage of coal particles <75µm**			
Grinding time (min)	**0**	**5**	**20**	**45**
F-1	0.1	2.0	5.4	8.7
F-2	0.4	12.7	39.7	80.8
F-3	0.3	19.1	44.0	81.1
F-4	5.3	15.3	36.4	67.6
F-5	0.4	8.0	26.9	35.3
F-6	0.8	9.5	20.6	34.7
F-7	0.5	6.8	21.1	38.6
F-8	0.8	8.3	18.9	29.4

Comparison of data in Table 7.5 (microwave treated coals) and Table 7.4 (reference coals) show that the degree of fineness is increased as a result of microwave treatment prior to grinding. Microwave conditions are described in Section 7.5. In particular, the weight percentage of coal less than 75µm increases after irradiation, (following 45 minutes grinding time) more so than for low rank coal. However, there is no change in the weight percentage less than 75µm for F-1 coal (anthracite) suggesting that microwave treatment had no effect upon the grindability of this coal.

Table 7.5 The degree of fineness after microwave treatment

Coal sample	**Weight percentage of coal particles <75µm**			
Grinding time (min)	**0**	**5**	**20**	**45**
F-1	0.1	1.2	4.4	8.4
F-2	0.4	15.6	48.9	84.2
F-3	0.3	16.9	69.7	88.7
F-4	5.3	19.5	46.7	83.6
F-5	0.4	13.7	39.4	86.9
F-6	0.8	11.8	34.1	50.4
F-7	0.5	8.1	29.3	66.4
F-8	0.8	14.1	29.0	72.2

The results also show that, for some medium and low rank coals, the volume of coal; milled to PF size has doubled in these batch samples (F-5) because of microwave exposure.

Further work would be necessary to investigate the effect shown by batch rod milling on fines production with regard to continuous air-swept ring ball mills.

7.8 The influence of microwave radiation on the proximate analysis of coal

Table 7.6 shows the proximate analyses for group 1 coals before and after microwave treatment (at 0.65kW power input and a frequency of 2.45GHz for 8 minutes).

Due to the heterogeneous nature of coal the before and after irradiation, ash contents are all within accepted experimental error range and should not be considered significant. More specifically, the moisture and volatile contents of the high rank coals (F-1, F-2 and F-3) were unaffected by irradiation. In contrast, the moisture content has been reduced on all other microwaved coals-showing evidence of drying which is particularly significant with low rank coals. Similarly, coal volatile content decreased on microwave treated coals, with the effect again being particularly marked on the low rank coals. Grindability studies on group 1 coals also indicate that low rank coals are particularly sensitive to microwave radiation. All results given, are mean values and within accepted standard deviations (Table 7.6, in brackets) as outlined in accord with BS1016.

It can be suggested that high-pressure gas release arising from the rapid heating of inherent moisture and volatile matter may be possible mechanisms for fracture propagation in the coal. This would be consistent with the fact that low rank coals contain larger quantities of inherent moisture and volatile matter and exhibit the greatest improvement in coal grindability following microwave treatment.

Table 7.6 Proximate analysis (as-received basis unless stated) of group 1 coals before and after microwave exposure

Proximate analysis for reference samples					
Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
F-1	1.6 (0.08)	1.8 (0.03)	4.6 (0.30)	92.0	4.8
F-2	0.8 (0.06)	4.0 (0.01)	12.7 (0.74)	82.5	13.4
F-3	1.0 (0.02)	3.8 (0.04)	28.5 (0.65)	67.7	29.8
F-4	5.8 (0.03)	5.2 (0.07)	33.0 (1.65)	56.0	37.6
F-5	6.1 (0.06)	3.4 (0.05)	34.8 (0.24)	55.7	38.7
F-6	6.1 (0.09)	4.4 (0.13)	35.7 (0.40)	53.8	40.4
F-7	4.7 (0.04)	5.0 (0.02)	37.1 (0.54)	53.2	41.6
F-8	13.3 (0.02)	7.4 (0.07)	34.9 (0.07)	44.4	45.1
Proximate analysis for samples exposed to 0.65kW, 2.45GHz for 8 minutes					
F-1	1.4 (0.09)	1.4 (0.25)	4.7 (0.62)	92.5	4.9
F-2	0.7 (0.04)	3.8 (0.11)	10.4 (0.17)	85.1	10.9
F-3	0.6 (0.09)	3.4 (0.01)	25.9 (0.47)	70.1	27.0
F-4	1.2 (0.19)	4.6 (0.10)	32.4 (0.58)	61.8	34.4
F-5	0.8 (0.25)	3.3 (0.06)	30.7 (0.48)	65.2	32.0
F-6	1.0 (0.17)	3.7 (0.06)	34.2 (0.57)	61.1	35.9
F-7	0.7 (0.22)	4.8 (0.00)	34.9 (0.43)	59.6	37.0
F-8	1.7 (0.26)	10.2 (0.00)	34.4 (1.24)	53.7	39.0

7.9 Ultimate analysis of microwave exposed coals

Ultimate analysis (dry, mineral matter free basis) of group 1 coals before and after microwave treatment have also been compared (Table 7.7). The analyses were expressed on a dry, mineral matter free basis and specifically determine the major elemental constituents of the organic coal component. Prior to microwave treatment, these coals, collectively, show that as coal rank decreases the carbon content decreases (90% for F-1 to 63.5% for F-8). Hydrogen and nitrogen remain relatively constant whilst the sulphur content increases slightly (0.6% for F-1 to 2% for F-8). The oxygen content increases with decreasing coal rank indicating the presence of increasing volatile matter content with low rank coals.

Table 7.7 Ultimate analysis (dry, mineral matter free basis) of group 1 coals before and after microwave treatment

Ultimate analysis for reference samples					
Coal sample	Sulphur %	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
F-1	0.57	90.10	2.43	1.10	5.80
F-2	0.66	88.40	3.66	1.50	5.78
F-3	1.06	83.50	4.60	1.30	9.54
F-4	2.12	73.80	4.56	1.80	17.72
F-5	1.70	74.80	4.43	1.70	17.37
F-6	1.43	71.70	4.30	1.30	21.27
F-7	1.39	76.00	4.96	1.90	15.75
F-8	1.98	63.50	3.95	1.50	29.07
Ultimate analysis for samples exposed to 0.65kW, 2.45GHz for 8 minutes					
F-1	0.61	90.40	2.43	1.10	5.46
F-2	0.64	87.80	3.70	1.50	6.36
F-3	1.07	83.00	4.60	1.40	9.93
F-4	2.17	75.00	4.58	1.70	16.55
F-5	1.70	75.80	4.70	1.70	16.10
F-6	1.40	72.60	4.35	1.30	20.35
F-7	1.43	76.10	4.99	1.90	15.58
F-8	2.55	64.50	4.04	1.50	27.41

Ultimate analyses of the microwave-treated coals show negligible changes between sulphur, nitrogen and hydrogen. However, some slight differences occur between the carbon and oxygen content of low rank coal (F-8). The oxygen content has reduced from 29% to 27.5% and a subsequent increase of 1% in carbon content for the microwave treated coal. Whilst these differences are small, they may reflect the changes possible due to the reduction of volatile and carbon content following heating as indicated in the proximate analyses (Table 7.6). The ultimate analyses showed that there was little change in the coal following microwave treatment; this feature implies that there are properties of coal (e.g. moisture and mineral content) which may predominantly contribute to the microwave grindability effect.

7.10 The effect of muffle furnace drying on coal rank grindability

Additional samples of F-6 and F-7 coals were conventionally heated prior to milling to examine whether or not the microwave grindability effects (Table 7.3) could be duplicated by convective thermal drying. These particular coals were chosen in so far as they showed significant improvements in grindability with microwave exposure. Milling conditions were identical to those for the microwaved coals (Section 7.6), and before milling, the coals were exposed to conventional heating for one hour to ensure their bulk temperature was in equilibrium with the furnace temperature.

Table 7.8 Relative coal (F-6) grindability effects after muffle furnace and microwave heating

Coal	Relative Work Index %		
Grinding time (minutes)	**5**	**20**	**45**
F-6 non-treated	100	100	100
F-6 (muffle furnace) 100°C	69	60	66
F-6 (muffle furnace) 200°C	69	72	63
F-6 microwave treated (0.65kW 8 min)	72	74	74

Table 7.9 Relative coal (F-7) grindability effects after muffle furnace and microwave heating

Coal	Relative Work Index %		
Grinding time (minutes)	**5**	**20**	**45**
F-7 non-treated	100	100	100
F-7 (muffle furnace) 100°C	69	78	64
F-7 (muffle furnace) 200°C	49	60	57
F-7 microwave treated (0.65kW 8 min)	68	69	61

The coals were heated at furnace temperatures of 100°C and 200°C and the size distributions following milling were expressed in terms of the Relative Work Index. The results (Table 7.8, Table 7.9) show that conventional heating also increases the grindability of these sub-bituminous coals. Similar results can be seen for both the F-6 furnace heated samples, suggesting the main mechanism responsible for improved grindability may be effected at lower temperatures for this particular coal. The grindability of F-7 samples increases above 200°C implying that, if the inherent moisture is responsible for this improvement, then temperatures above 100°C may be required for maximum beneficiation. The results also indicate as to what the temperatures achieved by microwave treatment where and also suggest that improvements may be increased by maintaining elevated temperatures for longer than was allowed for microwave treatment.

7.11 The effect of microwave exposure on coal rank calorific value

The calorific values of group 1 coals (reference and microwave treated samples) were determined in accordance with BS1016 part 5 (Appendix A). Results (Table 7.10) are mean average values taken from a series of measurements on each coal. Table 7.10 shows, as expected, a decrease in calorific value with decreasing coal rank.

Table 7.10 Effect of microwave radiation upon the calorific value (dry, mineral matter free basis) of group 1 coals

Coal	Calorific Value $(MJ/kg)_{dmmf}$		Percentage change (%)
	Non-treated coal	Microwave treated	
F-1	36.5	36.0	1.35
F-2	37.1	36.9	0.65
F-3	36.9	36.9	0.0
F-4	34.9	34.4	1.58
F-5	33.8	33.2	1.60
F-6	32.2	32.1	0.40
F-7	35.3	35.2	0.45
F-8	31.6	30.5	3.32

Table 7.10 also shows that microwave exposure has reduced the coal calorific value. However, the reduction in calorific value is small and possibly of significance only for F-8 coal. The loss in calorific value (dry, mineral free basis) probably occurs because of part of the volatile matter loss due to the relatively high temperaturse reached during microwave treatment. The calorific value was measured for a selected number of furnace-heated group 1 coals (F-6 and F-7) (Table 7.11). Results show, that at temperatures up to 100°C there is relatively no change in calorific value; however, at temperatures up to 200°C there is a small but significant reduction (<1%). It is noted that the calorific value of both F-7 and F-6 microwave-treated show only small changes, less than the samples conventionally-heated at 200°C. The implication is, that if microwave treatment is

optimised, coal grindability can be improved with no change to calorific value of some coals.

Table 7.11 Effect of muffle furnace and microwave heating on coal calorific value (dry, mineral matter free basis)

	Calorific Value (MJ/kg)$_{dmmf}$	
Coal	**F-6**	**F-7**
Non-treated	32.2	35.3
Furnace-heated, 100°C	32.8	35.3
Furnace-heated, 200°C	32.1	34.7
Microwave-heated (0.65kW, 8 min)	32.1	35.2

7.12 The effect of microwave-heating on coal specific surface

The specific surface (surface area per unit weight) of twelve coals (six group 1 coals and their corresponding microwave-treated equivalents) were determined using a 'Micromeritics Flow Sorb II 2300 surface area system'. Surface area measurements are made upon the nitrogen absorption technique. Each coal sample (500g) was pre-crushed to <0.2mm and size distributions maintained for standardisation of measurements (reduce particle shape irregularities). The sample was vacuum de-watered and purged with a measured volume of (30% nitrogen in helium) gas.

Table 7.12 The effect of microwave heating on coal specific surface

	Specific Surface Area m^2/g		Percentage change (%)
Coal sample	Non-treated	Microwave-heated	
F-2	3.39	3.72	+ 9.7
F-3	2.74	3.22	+ 17.5
F-4	3.87	4.22	+ 9.0
F-5	5.35	5.94	+ 11.0
F-6	2.33	2.86	+ 22.7
F-8	4.69	6.08	+ 29.6

Results (Table 7.12) show an increase in coal specific surface following microwave heating (0.65kW applied power input for 8 minutes). The increase in surface area after microwave treatment becomes most marketable on low rank coal. It is suggested that the increase in specific surface is related to fractures (Figure 7.4) caused by microwave heating (expansion of mineral inclusions and steam generation from inherent moisture).

7.13 Microwave grindability with coal rank conclusions

Following microwave treatment, grindability is most effective on low rank coal. One possible explanation for the results is that inherent moisture in the coal (which increases with reducing coal rank) may contribute (following steam generation) to weaknesses in the coal structure. A further feature is that flaws may also be produced from differential expansion of mineral inclusions (coals were chosen due to there low mineral content, to examine the effect of microwaves on the grindability of different rank coals). This could be beneficial as lignite coals are relatively difficult to grind in comparison to medium rank coal (Section 7.4). It was also noted that grindability was affected as a result of thermal heating and is not specific to microwave treatment. However, microwave treatment may improve grindability further depending upon the heating conditions and coal constitution (mineral inclusions and moisture content).

CHAPTER EIGHT

DIELECTRIC PROPERTIES OF COAL

8.1 Introduction-Dielectric properties of coal

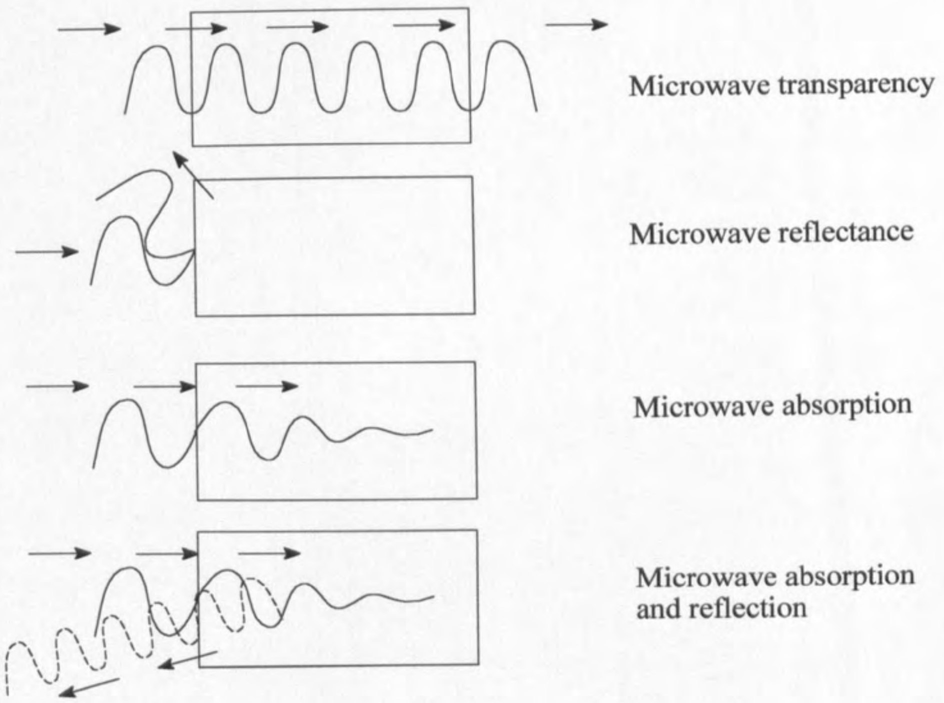


Figure 8.1 Behaviour of material within a microwave field (Church 1988)

Materials differ in their ability to absorb microwave radiation, some materials readily absorb microwaves whilst others appear transparent to or reflect microwaves (Figure 8.1). The aim of the study described below is to investigate to what extent coal is capable of absorbing microwave radiation. Preliminary studies (Chapter 6) have already demonstrated that (i) microwave radiation can improve the grindability of coal, and (ii) increased exposure to microwave radiation increases the overall bulk temperature of the coal. However, as the aim is to beneficially treat the coal, microwave exposure was limited such that a maximum sample temperature of between 200°C–250°C (to prevent de-volatilisation and ignition) was not exceeded

8.2 Measuring theory

Whilst a number of techniques exist for dielectric measurement (permittivity at a specific frequency), the circular resonant cavity method (as used in this study) is regarded as the most suitable for frequencies above 100MHz (Greenache 1996). More generally, frequencies above 600MHz are considered simpler to measure due to practical cavity size limitations i.e. at lower frequencies, the cavity diameter must be increased to accommodate resonant frequencies. The method used for dielectric measurement is based upon standard theoretical dielectric principles and measurements of the change in quality factor (Q-factor, Equation 8.1) and resonant frequency when the sample is inserted.

Measurements of specific resonant frequencies can be detected using a network analyser, in which measurements of the frequencies and their corresponding bandwidths are recorded. A quality factor, Q-factor is defined as:

$$Q = \omega \frac{\text{energy stored in the resonator dielectric}}{\text{power loss in theresonator (dielctric and cavity wdls)}} \tag{8.1}$$

It is possible to eliminate the effect of the loss through the walls with the equation:

$$\frac{1}{Q_1} - \frac{1}{Q_0} = \frac{\text{power loss in the dielectric}}{\omega \times \text{energy stored in the dielectric}} \tag{8.2}$$

In which, Q_0 the quality factor for the air-filled cavity and Q_1 is that for the material inserted into the cavity. The Q-factor is simply determined as the ratio of the resonant centre frequency to the resonant bandwidth.

This method can be used as a means of calibrating the dielectric properties of the inserted sample to the measured properties (due to air, cavity walls and sample holder) prior to the sample insertion. The left-hand side of Equation 8.2 is, by definition, equal to the loss tangent (Chapter 2) and can be optimally used to determine the imaginary part of the complex permittivity.

The real permittivity is evaluated from measurements of the change in resonant frequency following the sample introduction into the cavity and depends upon the sample to cavity volume ratio. The classical perturbation equation for the real component is given by Equation 8.3 (Greenache 1996).

$$\varepsilon' = 1 + 2J_1^2(x_{1,m})\frac{\delta f}{f_0}\frac{V_{cavity}}{V_{sample}} \tag{8.3}$$

Where.

ε'	Real permittivity (F/m)
V_{cavity}	Volume of cavity (m^2)
V_{sample}	Volume of sample (m^2)
f_0	Resonant centre frequency (air filled) (Hz)
δf	Change in centre frequency when sample is inserted (Hz)
$J_1(function)$	First order Bessel function (Perry 1984)
$(x_{1,m})$	Function of the air filled cavity equated by Equation 7.4 (Greenache 1996)

$$(x_{1,m}) = \frac{2\pi f_0 r_{cavity}}{c} \tag{8.4}$$

r_{cavity} Radius of the resonant cavity (m)

The classical perturbation equation for the imaginary component is given by Equation 8.5 (Greenache 1996).

$$\varepsilon'' = J_1^2(x_{1,m})\left(\frac{1}{\mathrm{Q}_1} - \frac{1}{\mathrm{Q}_0}\right)\frac{V_{cavity}}{V_{sample}} \tag{8.5}$$

Where.

ε'' Imaginary permittivity (F/m)

8.3 Dielectric measuring method

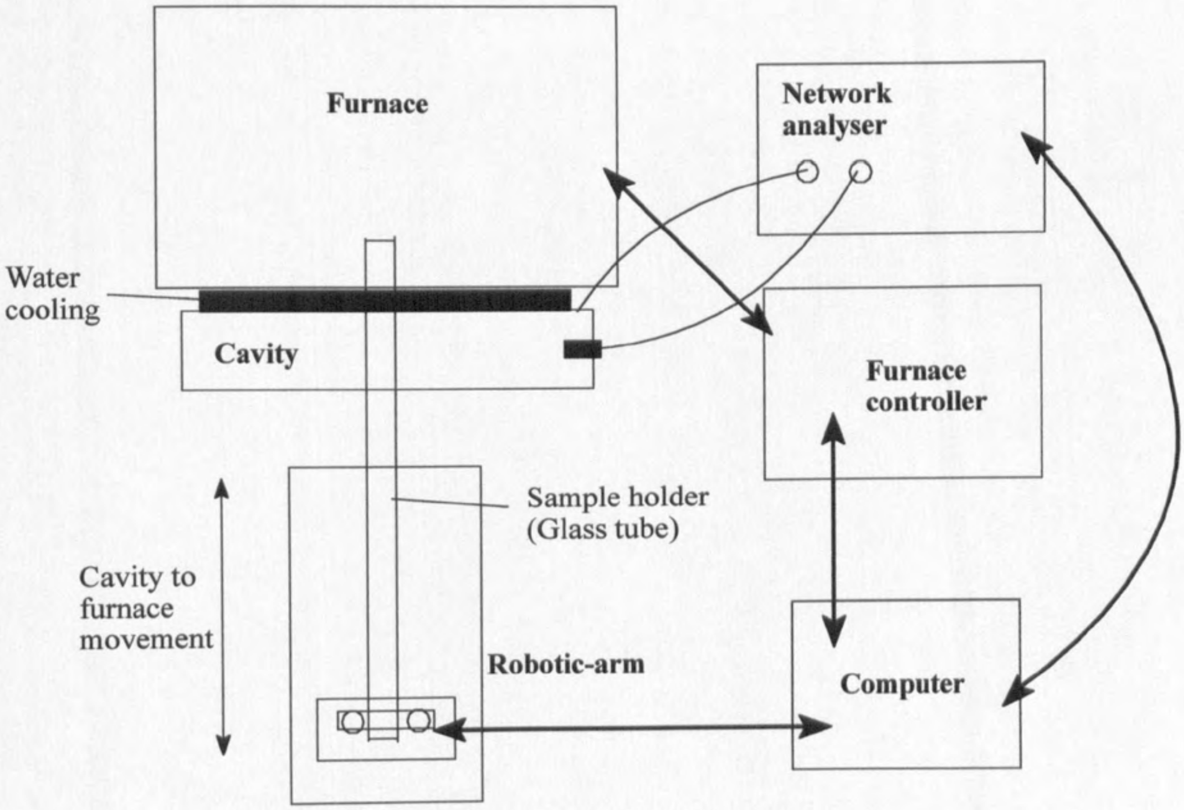


Figure 8.2 Diagrammatic representation of the dielectric measuring system (Greenache 1996)

A resonant cavity method was employed to measure the dielectric properties of coal. The experimental system (Figure 8.2) consists of two main components (i) a resonant cavity constructed of an appropriate material (copper) and of specific dimensions to produce resonant frequencies and (ii) a furnace to heat the sample to desired temperatures. The use of copper (as opposed to aluminium) cavity manufacture is considered to provide the most accurate measurements compared to theoretical resonant frequencies (Table 8.1) (Greenache 1996). The specific resonant frequencies produced depend upon both the cavity dimensions and the material of construction. The dimensions of the cavity in current use are shown in Figure 8.3 and the cavity resonant frequencies are shown in Table 8.1. A 'Hewlett Packard 8753C' vector network analyser, capable of operating between the frequency range 300kHz to 6GHz was used to transmit electromagnetic wave transmission and the detection of frequency shift.

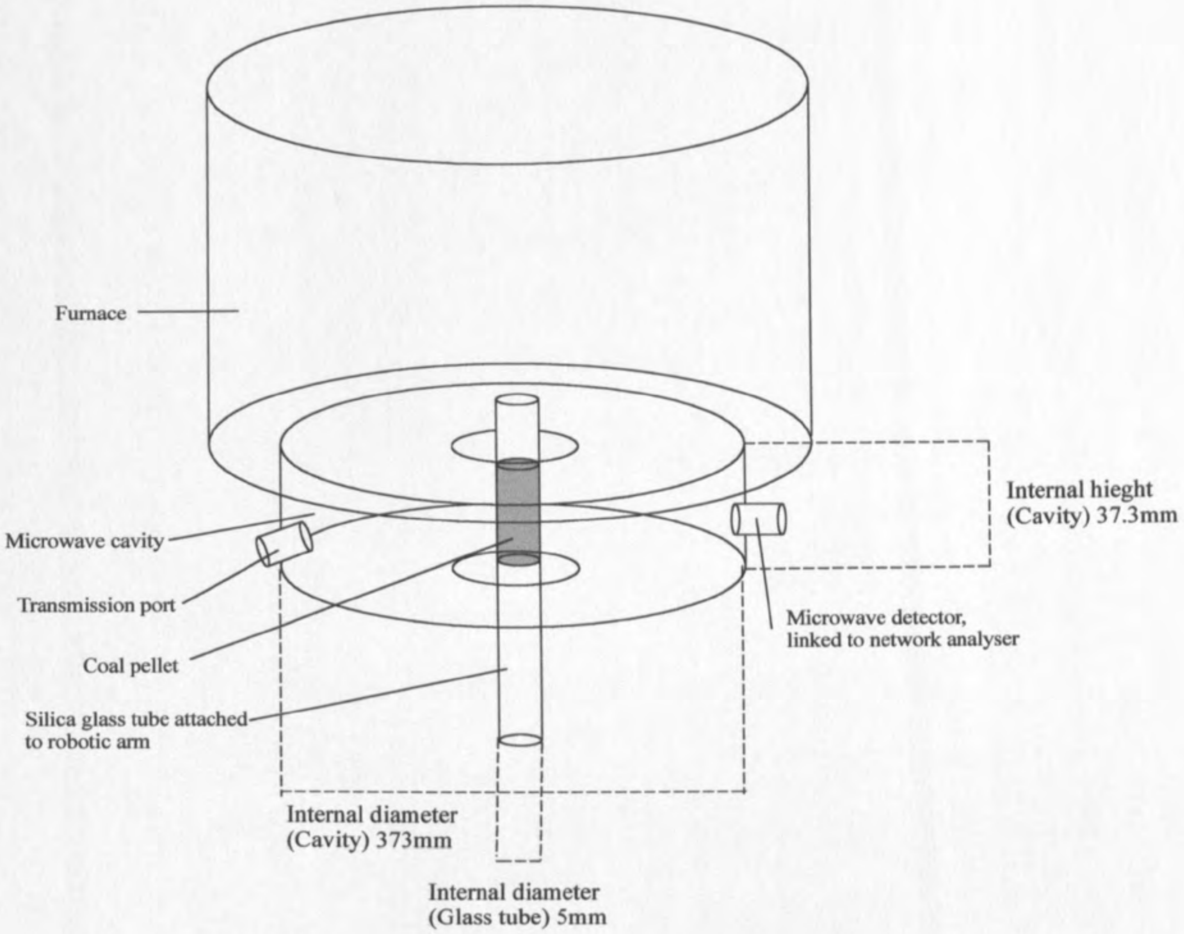


Figure 8.3 Microwave resonant cavity test equipment

A 'Carbolite' tube furnace capable of operating at 1800°C maximum temperature was used to maintain the temperature within the sample. Silica tube sample holders are mounted on a computer-operated stepper motion controller, the time the sample spends outside the furnace is a sum of the travelling time (<1s) and the response time of the network analyser (<15s). The silica holders (approximately transparent to microwaves) have been calibrated and this feature is incorporated into the computer-automated program. A water cooling system is attached between the furnace and resonant cavity to prevent cavity distortion at elevated temperatures. A 'Visual Basic' program, customised by the University of Nottingham ultimately controls the system.

Table 8.1 Measured and theoretical resonant frequencies of the dielectric measurement system (Greenache 1996)

Standard resonant Mode	Aluminium cavity resonant frequency (GHz)	Copper cavity resonant frequency (GHz)	Theoretical resonant frequency (GHz)
TM010	0.6091	0.6154	0.6152
TM020	1.3987	1.4127	1.4122
TM030	2.1938	2.2160	2.2139

A compressed pellet of coal powder, encased within a silica glass tube was injected into the microwave resonance cavity. Dielectric properties at room temperature where first measured by a computerised network analyser at three different frequencies (0.615, 1.413 and 2.216GHz) selected as they are resonant frequencies close to the working frequencies (650MHz and 2,450MHz) currently used in most domestic and industrial microwave units. The Bessel function (Equation 8.6) for the air-filled cavity at these frequencies are given in Table 8.2.

Table 8.2 Calculated first-order Bessel function for dielectric measurement

Standard resonant Mode	Copper cavity resonant frequency (GHz)	$x_{1,m}$ (Equation 7.4)	$J_1(x_{1,m})$
TM010	0.6154	2.40483	0.51915
TM020	1.4127	5.52008	-0.34026
TM030	2.2160	8.65373	0.27145

The coal sample was then mechanically positioned within the furnace above the cavity and the furnace temperature was increased at a rate of 1°C per minute. The temperature was maintained for 5 minutes to stabilise the temperature field within the coal pellet before the dielectric measurements were taken. The dielectric permittivities of the coal samples were measured between 40°C to 180°C at 20°C intervals.

$$J_1(x)=\left(\frac{x}{2}\right)^p \sum_{k=0}^{\infty} \frac{(-1)^k \left(x/_2\right)^{2k}}{k!(k+p)!} \tag{8.6}$$

Bessel function for integer values of 'P' (Perry 1984)
p denotes the order of the function (in this application $P = 1$)

8.4 The variation in dielectric properties of coal with increasing temperatures

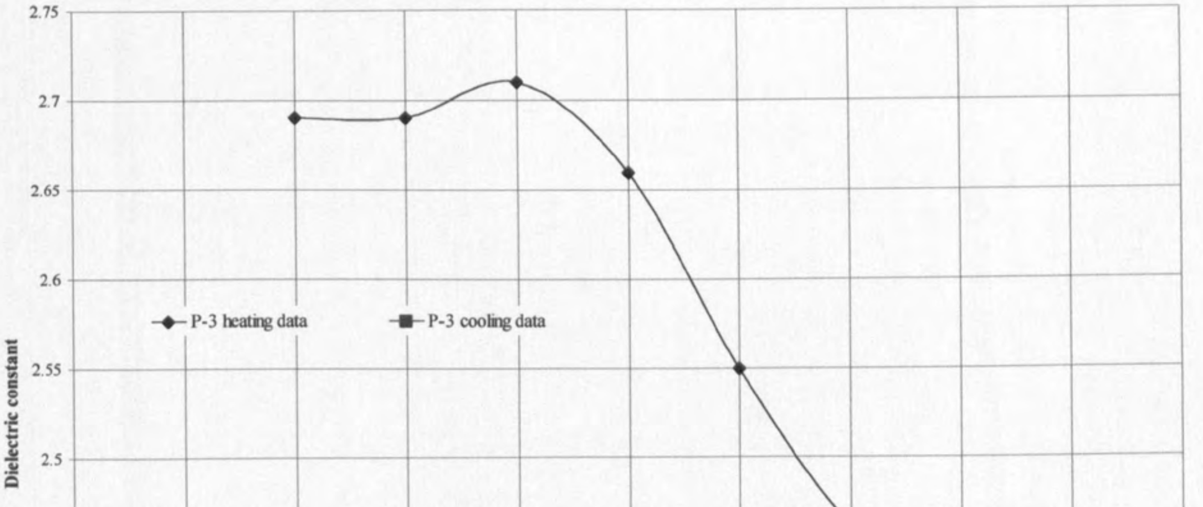


Figure 8.4 Importance of temperature and moisture content to coal (P-3) dielectric properties

The dielectric properties of both group 1 and group 2 coals were measured within the temperature range (40°C-180°C) similar to those reached in previous microwave heating trials (Chapter 6). Measurements were recorded at 20°C intervals during both the heating and cooling cycles. Figure 8.4 shows the dielectric constant variation with temperature; the corresponding loss factors (Appendix E.1) exhibit a similar trend upon heating and cooling. A substantial decrease in the dielectric constant is evident between 80°C and 180°C (heating cycle); in contrast, the dielectric constant remains relatively constant during the cooling cycle. This would suggest that, whatever accounts for the decrease in dielectric constant; upon heating, is either removed or irreversibly altered as a result of the temperature increase. The decrease in dielectric constant appears to correspond to the temperature range associated with the drying of coal. Coal moisture would decrease over such a temperature range. This would account for the subsequent reduction in both the dielectric constant and loss factor and would not be detected during the cooling cycle. All coals tested have shown the same behavioural characteristics, to varying degrees, irrespective of frequency.

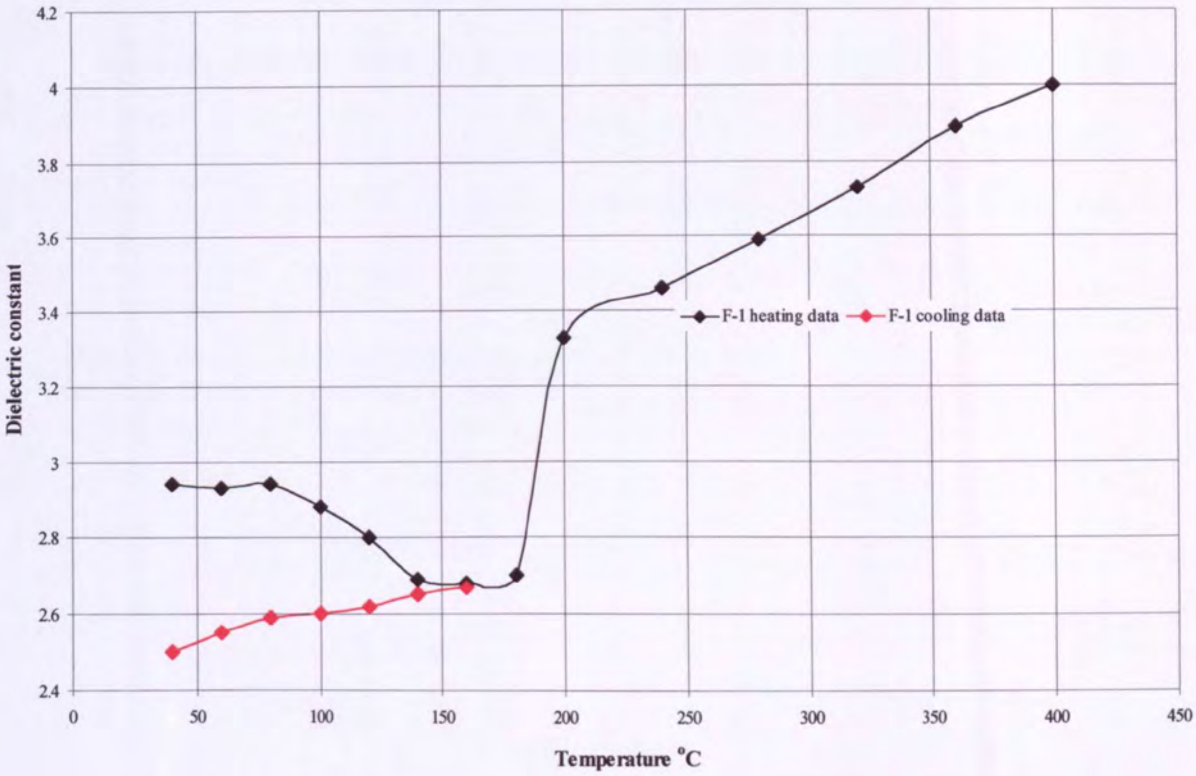


Figure 8.5 High temperature coal (F-1) dielectric measurement

The moisture reduction (drying) would affect the volume, voidage and bulk density of the test coal. However, as the moisture content represents a small fraction of the coal, the changes in sample condition (structure) at temperatures of up to approximately 180°C are believed to have little significance on the accuracy of the measurements. At higher temperatures coal de-volatilisation would be expected to have a marked effect upon the bulk density of the sample and this may subsequently reduce the accuracy of the measurement. To minimise any potential inaccuracies due to gaseous release, high temperature dielectric measurement was only attempted on a low-volatile coal (F-1). Measurements were made on two samples of this coal, the first being heated to180°C and allowed to cool, the second heated to 400°C. The initial stages of heating produced similar results (40°C-180°C) to those previously observed with other coals. However, a relatively large increase in dielectric constant (and loss factor) was measured between 180°C and 200°C followed by further increases in dielectric properties with temperature. This may be as a result of changes in coal bulk density. However, the results indicate that a ability of a dry coal to absorb microwave radiation may increase with temperature.

8.5 Dielectric properties of coal at selected frequencies

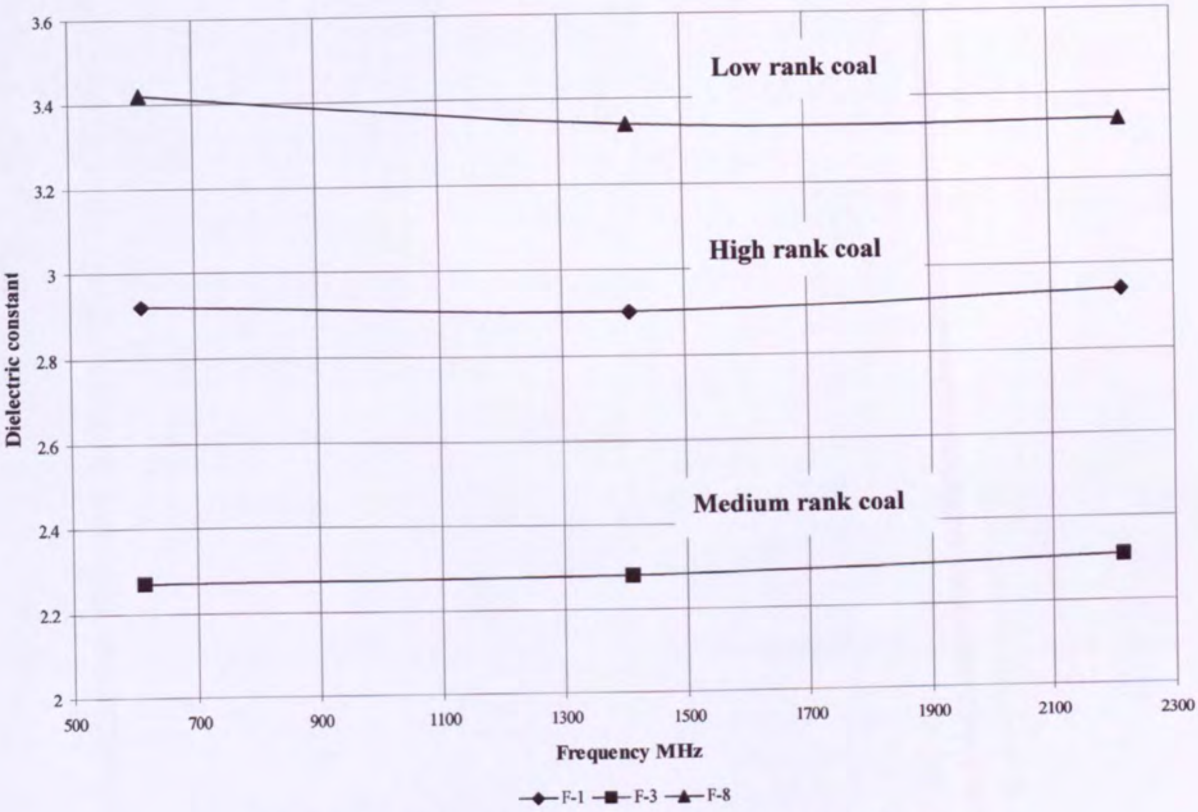


Figure 8.6 Coal dielectric constant dependence on frequency

Dielectric measurements carried out at three different frequencies (Figure 8.6) show that there are no common changes in dielectric properties with frequency. Other workers (Harrison 1997) have shown that the dielectric properties of coal can vary with frequency. However, the current results show that the change is small and hence not considered a major factor influencing the microwave absorption capability of the coal.

The variation in frequency may be affected by coal chemistry, particularly rank and mineral matter composition and content.

8.6 The variation of dielectric constant with coal rank

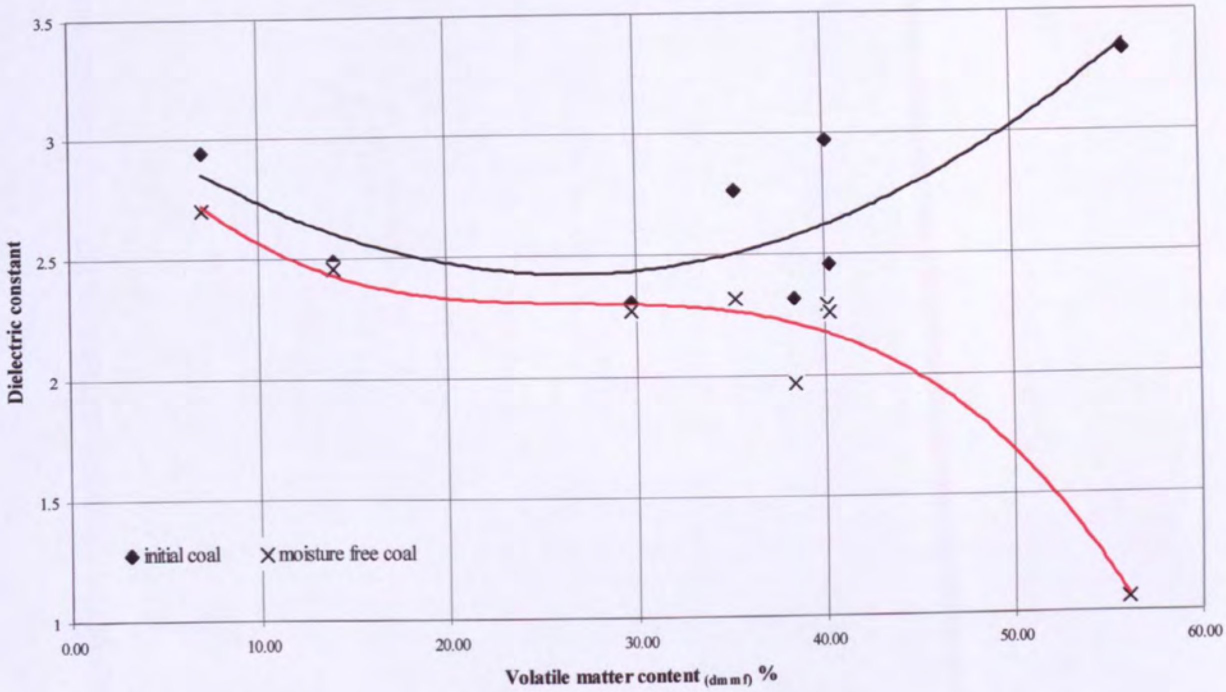


Figure 8.7 Variation of dielectric constant with coal rank

The chemical composition of the coal will ultimately affect its capability to absorb microwave radiation. Moisture, volatile matter and fixed carbon content vary according to rank, however, the mineral matter content and composition depends upon geological age of the coal and its proximity to seam boundaries. To determine the dielectric properties on coal rank, group 1 coals of various ranks were selected. All coals exhibit low mineral matter contents.

The dielectric properties of group 1 coals before and after drying are shown in Figure 8.7. Prior to drying the highest dielectric constants and therefore the greatest absorption capabilities are attributable to low (F-8 to F-6) and high (F-1 to F-3) rank coals. Medium ranked coals exhibit lower dielectric constant values. After drying, the low rank coals exhibit the lowest dielectric constant and account for the greatest change in dielectric property due to moisture removal. Medium rank coals show relatively similar dielectric constant values whilst high ranking coals show the least change due to drying and the highest values. This implies that the moisture content significantly increases the coals dielectric constant and microwave absorption ability, however, dielectric constants and loss factor increase with increasing rank on a dry basis. This suggests that dielectric properties are influenced by coal structure, higher absorption capabilities with increased fixed carbon to volatile matter contents.

In contrast, group 2 coals exhibit no common dielectric characteristics with relation to carbon content (rank). A possible explanation is that the significant variations are due to the coal mineral matter content and composition, which mask the coal rank effect.

8.7 Penetration depth of microwave radiation

The penetration depth is defined as the distance from the surface of the material at which the power reduces to the number e^{-1} from the value at the surface. The penetration depth has been derived from Maxwell's wave equation of the electromagnetic field in the z direction (Figure 8.8, Von Hippel 1954).

For simplicity, some penetration depth approximations, in particular Equation 2.11 assumes negligible changes due to the magnetic part of the wave equation. Accordingly, the complex permittivity and field electrical properties have a controlled effect on the penetration depth.

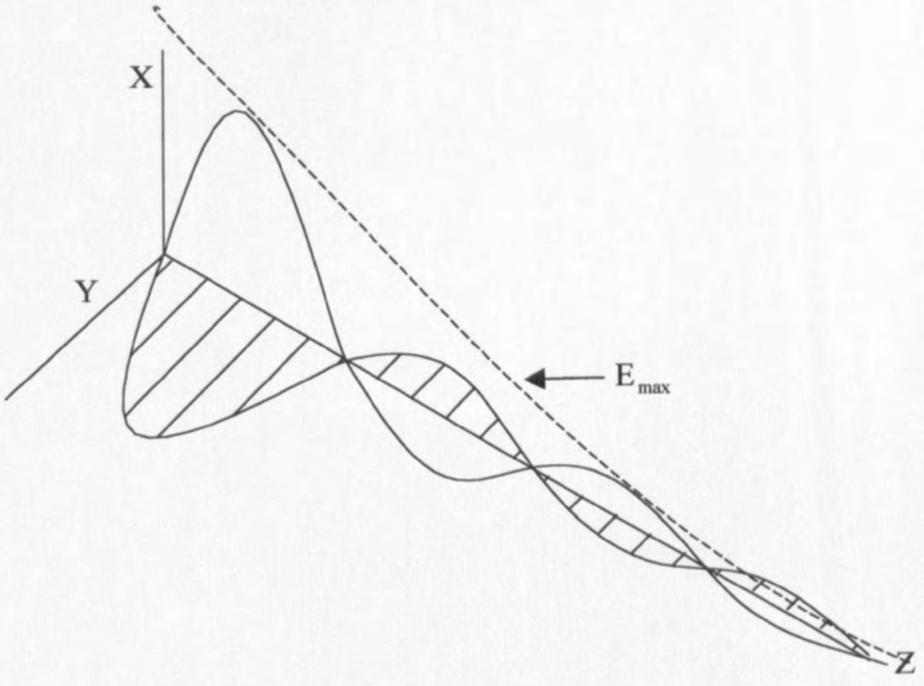


Figure 8.8 Propagation of a plane wave in a lossy material (z direction) (Metaxas 1983)

For low loss materials ($\varepsilon''/\varepsilon' < 1$) the penetration depth can be approximated (Equation 2.11, Metaxas 1983).

$$D_p = \frac{\lambda . \varepsilon_r'^{1/2}}{2\pi . \varepsilon_r''.} \qquad (2.11)$$

D_p	Penetration depth of wave (m)
ε_r	Relative dielectric constant or relative real permittivity
λ	Wavelength (m) $\lambda = c/f$
ε_r''	Relative loss factor or relative imaginary permittivity

In addition to penetration depth, skin depth is defined as that distance from the surface were the field absorbed decreases to e^{-1}. Derivations of the skin depth have previously been reported based upon the wave equation (Equation 8.7, Florek 1995).

$$D_s = \frac{c}{2\pi f}\sqrt{\frac{2}{\left(\varepsilon_r'^2+\varepsilon_r''^2\right)^{1/2}-\varepsilon'}} \qquad (8.7)$$

c Speed of light (m/s) 3×10^8 m/s

The measured penetration and corresponding skin depths of group 1 coals (Equation 2.11 and 8.7 respectively) are given at a frequency of 2.216GHz in Tables 8.3 and 8.4 respectively.

Table 8.3 Microwave penetration depth for group 1 coals (Metaxas 1983)

Coal	Dielectric constant (dimensionless)		Loss factor (dimensionless)		Penetration depth (m)	
	ε_r' 60 °C	ε_r' 180 °C	ε_r'' 60 °C	ε_r'' 180 °C	D_p 60 °C	D_p 180 °C
F-1	2.93	2.70	0.1657	0.1661	0.223	0.213
F-2	2.49	2.46	0.0539	0.0406	0.631	0.833
F-3	2.34	2.27	0.0818	0.0696	0.403	0.467
F-4	2.77	2.32	0.2087	0.0634	0.172	0.518
F-5	2.32	1.97	0.1389	0.0346	0.236	0.875
F-6	2.86	2.29	0.1564	0.0579	0.233	0.564
F-7	2.45	2.26	0.1252	0.0618	0.270	0.525
F-8	3.51	1.06	0.3153	0.0037	0.128	6.001

Table 8.4 Microwave skin depth for group 1 coals (Florek 1995)

Coal	Dielectric constant (dimensionless)		Loss factor (dimensionless)		Skin depth (m)	
	ε_r' 60 °C	ε_r' 180 °C	ε_r'' 60 °C	ε_r'' 180 °C	D_s 60 °C	D_s 180 °C
F-1	2.93	2.70	0.1657	0.1661	0.446	0.427
F-2	2.49	2.46	0.0539	0.0406	1.263	1.666
F-3	2.34	2.27	0.0818	0.0696	0.807	0.934
F-4	2.77	2.32	0.2087	0.0634	0.344	1.036
F-5	2.32	1.97	0.1389	0.0346	0.473	1.750
F-6	2.86	2.29	0.1564	0.0579	0.467	1.127
F-7	2.45	2.26	0.1252	0.0618	0.539	1.049
F-8	3.51	1.06	0.3153	0.0037	0.257	12.002

From Tables 8.3 and 8.4 it is apparent that the penetration depth is approximately half that of the skin depth, this is an indication of the consistency of Equations 2.11 and 8.7 with the wave equation (Cross 2000). Assuming no change in power absorption due to the magnetic component of the wave equation, the power absorbed by a material is derived from Equation 2.7. Power absorbed is proportional to (electric field strength)^2, and it is therefore expected that as part of the derivation $(\delta f x^2 = 2x)$, skin depth is approximately equal to 2(penetration depth). Accordingly, it is evident that any trend observed with penetration depth will also be observed upon the skin depth. For this reason, further discussion concerns the penetration depth only.

From Equation 2.11, it is seen that the penetration depth varies with rank and moisture content (temperature). With the exception of F-1 coal, penetration depth decreases from 0.6m (medium rank) to 0.1m (low rank). However, data for dry coals at 180°C show that low rank coal exhibits the greatest penetration whilst there is little change in the high rank coals.

The penetration depth generally increases at higher temperatures (Table 8.3) and it is expected that the coal would be moisture free at 180°C. Water has a penetration depth of approximately 25mm at 60°C, and the depth increases with temperature (Metaxas 1983). Hence, coal penetration depth will depend upon both the moisture content and temperature. Figure 8.9 shows that the penetration depth increases with temperature upon the heating cycle, but remains relatively constant on the cooling cycle. This could occur as a result of steam generation and evolution (moisture removal).

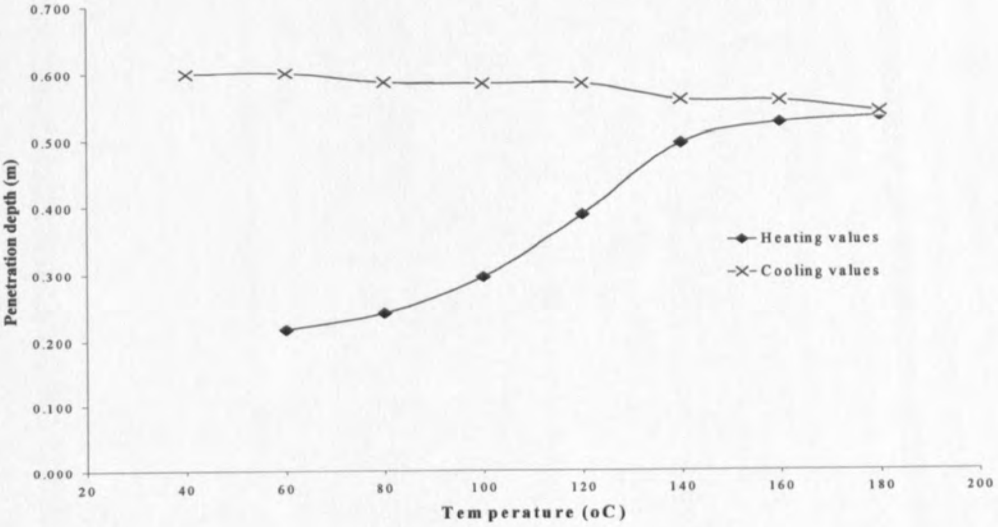


Figure 8.9 Variation of microwave penetration depth with temperature for F-8 coal

Penetration depth is significantly affected by frequency and decreases with increase in frequency (Figure 8.10). This is due to the change in wavelength (corresponding to the frequency change) as detailed in to Equation 2.11.

It is noted that Figure 8.9 and Figure 8.10 were based upon Equation 2.11 only, as the characteristics depend upon dielectric properties, similar trends would be observed using Equation 8.7.

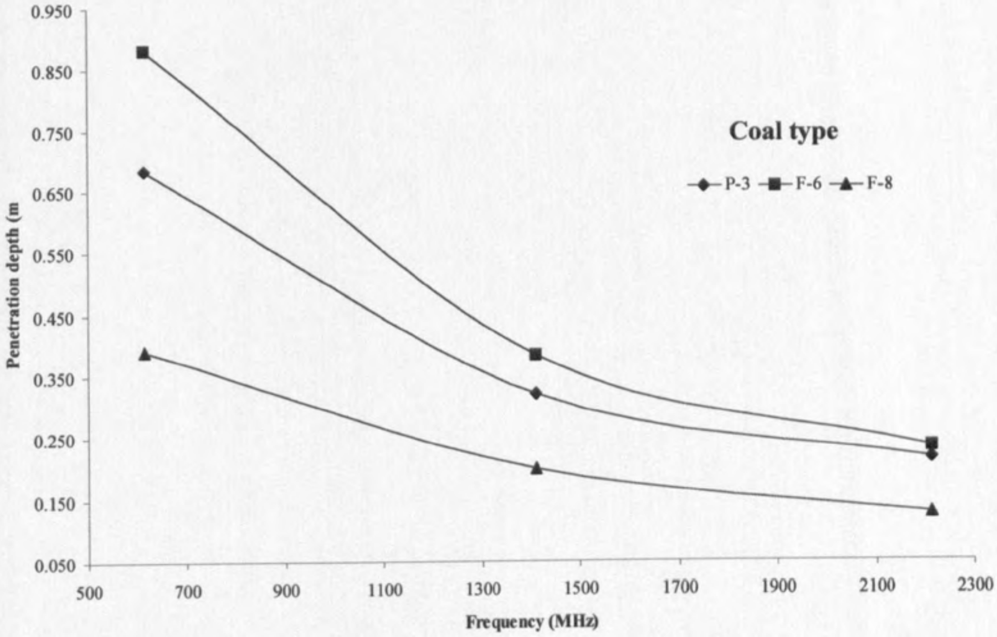


Figure 8.10 Variation of microwave penetration depth with applied microwave frequency

8.8 Coefficient of absorption and conductivity

8.8.1 Coefficient of absorption

The coefficient of absorption (A) is defined as the ability of a material to absorb microwave radiation and can be derived from the wave equation (Florek 1995, Equation 8.8).

$$A = \frac{f.\varepsilon''.E_{\text{insitu}}^2}{E^2} \qquad (8.8)$$

Where.

A	Coefficient of absorption (F/m^2)
ε''	Imaginary permittivity (F/m)
E	Electric field strength (v/m)
E_{insitu}	Electric field strength inside material (v/m)

E_{insitu}/E by definition is the coefficient of reflection (derivation of 'Snell's law and Fresnel's equations) (Von Hippel 1954), and represents the relationship between the electric field absorbed by the material to that of the cavity. The coefficient of reflection is unique to a given material and is used for theoretical heating rate calculations, which are discussed in further detail in subsequent sections. There have been many approximations for the relationship between cavity and material electric field strength i.e. Equation 8.9 (Mayer 1983).

$$\frac{E_{insitu}}{E} = \frac{3\varepsilon_r'}{\left(2\varepsilon_r' + 1\right)} = r_e \qquad (8.9)$$

ε_r'	Dielectric constant
E	Electric field strength (v/m)
E_{insitu}	Electric field strength within the material (average) (v/m)
r_e	Coefficient of reflection

However, Equation 8.9 is considered a crude approximation (Mayer 1983) and a more accurate method of deriving the coefficient of absorption (Equation 8.10) incorporates the particle size and shape (assumed spherical). The coefficient of absorption is by definition the fraction of incident energy not reflected, as such, the coefficient of absorption is (1-coefficient of reflection). It is claimed the most accurate relationship is that given by Equation 8.10 (Metaxas 1983).

$$A = \left(1 - e^{-2r/D_s}\right) \qquad (8.10)$$

r	Radius of particle (m)
D_s	Skin depth (m)
A	Coefficient of absorption (F/m^2)
e	Napierian logarithm base approximately equal to 2.7182818285 (10dp)

Table 8.5 Change of coefficient of absorption with coal rank (untreated coals)

Coal	Dielectric constant (dimensionless)		Loss factor (dimensionless)		Coefficient of absorption (F/m^2)	
	$\varepsilon_r'\ _{60}\ ^{o}C$	$\varepsilon_r'\ _{180}\ ^{o}C$	$\varepsilon_r''\ _{60}\ ^{o}C$	$\varepsilon_r''\ _{180}\ ^{o}C$	$A_{60}\ ^{o}C$	$A_{180}\ ^{o}C$
F-1	2.93	2.70	0.1657	0.1661	0.049	0.040
F-2	2.49	2.46	0.0539	0.0406	0.017	0.012
F-3	2.34	2.27	0.0818	0.0696	0.027	0.021
F-4	2.77	2.32	0.2087	0.0634	0.062	0.019
F-5	2.32	1.97	0.1389	0.0346	0.046	0.011
F-6	2.86	2.29	0.1564	0.0579	0.046	0.018
F-7	2.45	2.26	0.1252	0.0618	0.040	0.019
F-8	3.51	1.06	0.3153	0.0037	0.083	0.002

The coefficient of absorption generally decreases with coal rank on a dry basis, however, no specific trend can be seen for the 'as-received' coal (Table 8.5). Clearly, there is a relationship between the penetration depth (which increases with the removal of moisture) and the coefficient of absorption which decreases with moisture removal. It is noted that no relationship can be observed between those coals that are affected the most in terms of grindability improvements and the absorption ability. All values of the coefficient of absorption given in Table 8.5 are for spherical particles of 0.01m diameter.

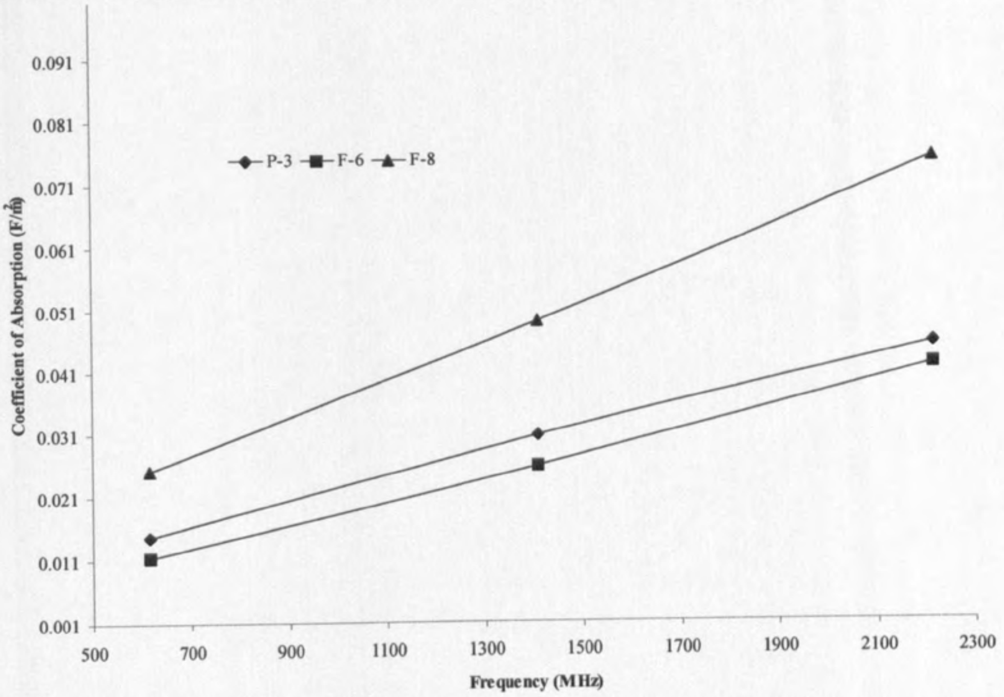


Figure 8.11 Variation of coefficient of absorption with microwave frequency

The coefficient of absorption also depends upon applied microwave frequency (Figure 8.11) and it is apparent that the shorter the wavelength, the greater the energy absorbed within a given material and hence, the greater the absorption capability.

8.8.2 Dielectric conductivity

The dielectric conductivity is considered relevant to dielectric heating because it is proportional to the power absorbed by the material (Equation 2.7).

$$\varepsilon'' = \frac{O_c}{\omega} \qquad (2.6)$$

O_c Dielectric conductivity (S/Ω)
ω Angular frequency (s^{-1})

$$P = O_c . \mathrm{E}_{\mathrm{insitu}}{}^2 \tag{2.7}$$

P	Power absorbed (W/m^3)
$\mathrm{E}_{\mathrm{insitu}}$	Electric field strength within material (v/m)

The dielectric conductivity can be evaluated from knowledge of the imaginary permittivity and the applied frequency of the radiation (Equation 8.11).

$$O_c = 2\pi f \varepsilon_r'' \varepsilon_o \tag{8.11}$$

f	Frequency (Hz)
ε_r''	Relative loss factor or relative imaginary permittivity
ε_0	Permittivity of free space 8.85×10^{-12} (F/m)

Table 8.6 Relationship between dielectric conductivity and coal rank at 60°C

Coal	**Loss factor at 60°C (dimensionless)**			**Dielectric Conductivity ($\Omega^{-1}m^{-1}$)**		
Frequency	ε_r'' 615MHz	ε_r'' 1413MHz	ε_r'' 2216MHz	O_c 615MHz	O_c 1413MHz	O_c 2216MHz
F-1	0.1630	0.1433	0.1657	0.005574	0.011259	0.020418
F-2	0.0443	0.0402	0.0539	0.001515	0.003159	0.006642
F-3	0.0543	0.0534	0.0818	0.001857	0.004196	0.010080
F-4	0.1951	0.1810	0.2087	0.006672	0.014221	0.025717
F-5	0.1381	0.1317	0.1389	0.004723	0.010348	0.017116
F-6	0.1494	0.1489	0.1564	0.005109	0.011699	0.019272
F-7	0.1150	0.1188	0.1252	0.003933	0.009334	0.015428
F-8	0.3792	0.3191	0.3153	0.012968	0.025072	0.038852

Table 8.7 Relationship between dielectric conductivity and coal rank at 180°C

Coal	**Loss factor at 180°C (dimensionless)**			**Dielectric Conductivity ($\Omega^{-1}m^{-1}$)**		
Frequency	ε_r'' 615MHz	ε_r'' 1413MHz	ε_r'' 2216MHz	O_c 615MHz	O_c 1413MHz	O_c 2216MHz
F-1	0.1934	0.1537	0.1661	0.006614	0.012076	0.020467
F-2	0.0336	0.0321	0.0406	0.001149	0.002522	0.005003
F-3	0.0375	0.0435	0.0696	0.001282	0.003418	0.008576
F-4	0.0387	0.0351	0.0634	0.001323	0.002758	0.007812
F-5	0.0259	0.0278	0.0346	0.000886	0.002184	0.004264
F-6	0.0484	0.0483	0.0579	0.001655	0.003795	0.007135
F-7	0.0496	0.0526	0.0618	0.001696	0.004133	0.007615
F-8	0.0035	-	0.0037	0.000120	-	0.000456

As both the coefficient of absorption and dielectric conductivity indicate the ability of a material to absorb microwave energy, it is expected the two parameters will show the same common trends in relation to frequency dependence, moisture and rank. Furthermore the power absorbed also mirrors this behaviour to some extent depending upon the applied electric field strength.

8.9 Dielectric properties of microwave treated P-3 coal

The dielectric properties of microwave treated (P-3) coal was measured. Coals (500g) of 3, 5 and 8 minutes irradiation at 0.65kW power (2.45GHz frequency) (Chapter 6.11) were sub-sampled after milling.

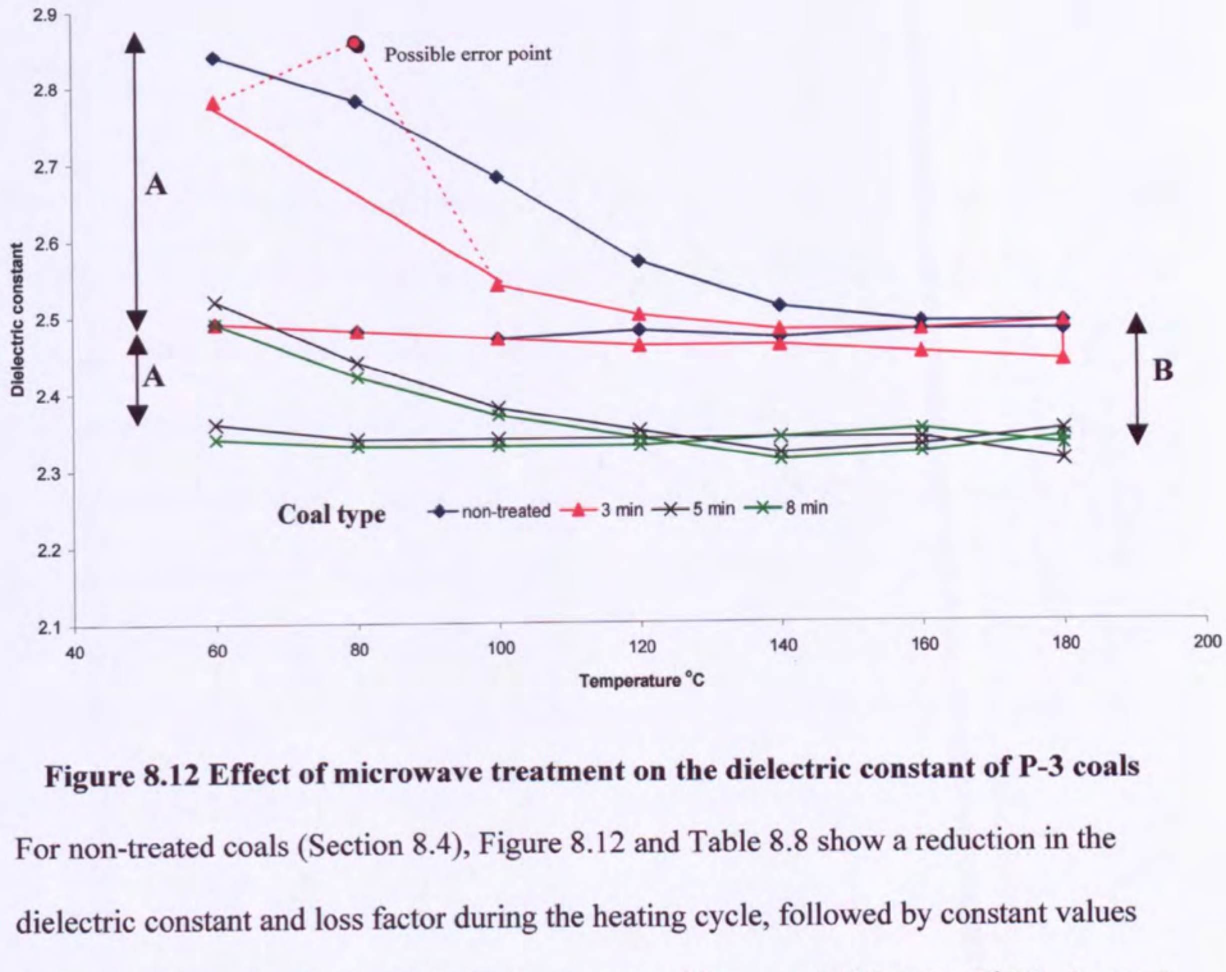


Figure 8.12 Effect of microwave treatment on the dielectric constant of P-3 coals

For non-treated coals (Section 8.4), Figure 8.12 and Table 8.8 show a reduction in the dielectric constant and loss factor during the heating cycle, followed by constant values on the cooling cycle (Section 8.4). Moisture removal between 80°C and 180°C is the most reasonable explanation (denoted by '**A**' on Figure 8.12). However, the initial values of dielectric constant (60°C), decrease with increasing microwave exposure times (Figure 8.12). This could be explained by previous steam generation due to microwave exposure, the moisture content reducing as microwave exposure times increase (Table 8.8).

Table 8.8 Dielectric properties, moisture and volatile matter contents of P-3 microwave exposed coals

Coal exposure time (minutes)	Dielectric constant (dimensionless)		Loss factor (dimensionless)		Moisture content (%)	Volatile Matter Content $_{dmmf}$ (%)
P-3 coal	ε_r' 60 °C	ε_r' 180 °C	ε_r'' 60 °C	ε_r'' 180 °C	M	VM_{dmmf}
0	2.84	2.49	0.1684	0.0639	4.70	38.86
3	2.78	2.49	0.1313	0.0576	2.98	41.67
5	2.52	2.35	0.0996	0.0491	2.98	40.68
8	2.49	2.34	0.0912	0.0453	2.26	34.93

It is also apparent that, after 5 minutes of exposure the cooling cycle values (constant) are lower than those determined for the non-treated and 3 minutes exposed coals (denoted by '**B**' on Figure 8.12). This may be due to a reduction in the volatile matter content of the coals exposed for 5 minutes and above (Table 8.8). In contrast, results discussed in Section 8.6, show that as volatile matter decreases (with moisture, in this case) the fixed carbon and mineral matter content (weight percentages) would increase, and hence the dielectric constant may be expected to increase. However, the mineral matter content of P-3 coal is 19.2%, (much higher than coals discussed in Section 8.6) and the mineral matter is predominately silicates and clay minerals which have a low microwave absorption ability. It is possible that, as the ratio of mineral matter content increases relative to the decrease in volatile matter, the dielectric constant values may decrease on a dry basis.

8.10 Dielectric properties of selected minerals and water

The dielectric properties of some selected (commonly coal associated) minerals were measured. Table 8.9 shows that, quartz and mica are relatively transparent to microwave radiation with relatively low coefficient of absorption and a high corresponding penetration depth. Calcite, dolomite and kaolin also exhibit low absorption and high penetration characteristics. Pyrite shows high absorption and low penetration, much higher absorption capabilities than any tested coal. Results (Table 8.9) show that each mineral has a unique heating behaviour upon microwave heating. The majority of minerals show a decreased coefficient of absorption value (possibly due to moisture). However, pyrite and (to a lesser extent) quartz give an increased coefficient of absorption.

Table 8.9 Dielectric properties of common coal associated minerals

Dielectric properties of selected minerals						
Mineral	**Pyrite**	**Quartz**	**Dolomite**	**Kaolin**	**Mica**	**Calcite**
$\varepsilon_r'{}_{60\,^{o}C}$	7.07	1.85	2.89	2.68	2.85	3.06
$\varepsilon_r'{}_{180\,^{o}C}$	7.58	1.99	2.97	2.56	2.83	-
$\varepsilon_r''{}_{60\,^{o}C}$	1.0625	0.0493	0.0465	0.1638	0.0403	0.0544
$\varepsilon_r''{}_{180\,^{o}C}$	1.2353	0.0695	0.0224	0.0104	0.0232	-
$D_{p\,60\,^{o}C}$ (m)	0.054	0.595	0.788	0.216	0.903	0.693
$D_{p\,180\,^{o}C}$ (m)	0.048	0.438	1.659	3.318	1.564	-
$A_{60\,^{o}C}$ (F/m^2)	0.185	0.019	0.014	0.050	0.012	0.016
$A_{180\,^{o}C}$ (F/m^2)	0.188	0.023	0.006	0.003	0.006	-

The dielectric properties are an indication of the heating characteristics of the selected minerals. All minerals tested (with the exception of pyrite) are expected to have a lower heating rate than a medium-ranked coal, pyrite is expected to heat at a much higher rate.

The cavity method was employed at the University of Nottingham to determine of the dielectric properties of water at different frequencies (Greenache 1996).

Table 8.10 Dielectric properties of Water (Greenache 1996)

Frequency (GHz)	ε_r' $_{25}$ °C (dimensionless)	ε_r'' $_{25}$ °C (dimensionless)	$D_{p\,25}$ °C (m)	A_{25} °C (F/m^2)
0.615	78.0	2.51	0.076	0.123
1.413	77.5	5.71	0.033	0.260
2.216	77.0	8.46	0.022	0.360

Similar trends were evident with regard to penetration depth and absorption (Table 8.10) and it is also noted that the dielectric constant and corresponding loss factor were much greater than those of the minerals tested. Penetration depths were also reduced and absorption ability was appreciably increased. The physical (gas-liquid-solid) phase undoubtedly has an effect upon the dielectric properties of materials. Liquids may exhibit much higher values of dielectric constant and loss factor than if they are present in the solid state i.e. ice at −12°C has a dielectric constant of 3.2 at a frequency of 3GHz (Von Hippel 1954).

8.11 Dielectric properties of dry, mineral free coal

Coals, by their nature, contain a variety of components-in the simplest sense inherent moisture, associated mineral matter (of different forms) and the organic coal substance. All of these constituents affect the dielectric properties of coals. Dielectric measurement on a dry, mineral matter free basis would depend upon de-mineralisation (without change to the organic coal component) and moisture removal. An alternative method of evaluating the organic coal (carbon content) dielectric properties is to use dielectric mixture equations.

The preferred mixture-equation (Masayuki 1995) for microwave frequency bandwidth is the Landau-Lifshifz formula (Equation 8.12):

$$\varepsilon^* = \left[\frac{\rho}{\rho_A} m_A (\varepsilon^*_A)^{1/3} + \frac{\rho}{\rho_B} m_B (\varepsilon^*_B)^{1/3} \right]^3 \tag{8.12}$$

m_x	Mass fraction of component *x*
ρ_x	Density of component *x* (kg/m^3)
ρ	Mean density of mixture (kg/m^3)
ε^*	Complex permittivity (replaced by the dielectric constant and loss factor respectively)

The formula can be presented for mixtures of more than two constituents providing the sum of the mass fractions is equal to unity.

$$\varepsilon^* = \left[\frac{\rho}{\rho_A} m_A (\varepsilon^*_A)^{1/3} + \frac{\rho}{\rho_B} m_B (\varepsilon^*_B)^{1/3} + \frac{\rho}{\rho_C} m_C (\varepsilon^*_C)^{1/3} \right]^3 \tag{8.13}$$

Given that the density of the individual minerals and organic coal component are known the bulk density can be evaluated (Equation 8.14, Nelson 1996).

$$\frac{1}{\rho_{bulk}} = \frac{m_A}{\rho_A} + \frac{m_B}{\rho_B} + \frac{m_C}{\rho_C} \tag{8.14}$$

8.11.1 Evaluation of dry, mineral free coal dielectric properties using the Landau-Lifshifz mixture equation

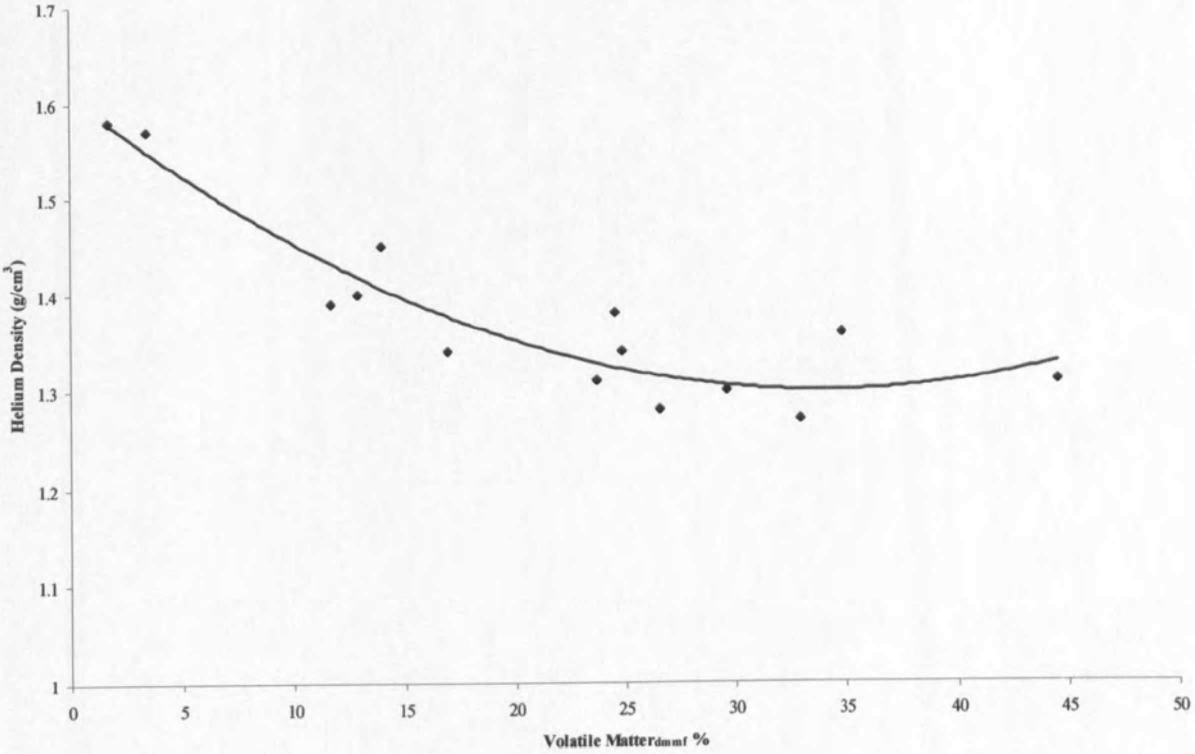


Figure 8.13 Helium density of various coals (dry, mineral matter free basis) (Elliot 1981)

The Landau-Lifshifz formula relates the dielectric properties of the components-and their proportions in the mixture-to the dielectric property of the mixture. The dielectric properties of the six most common minerals were measured using the resonant cavity method (Table 8.9). The corresponding densities of these minerals are shown in Table 8.14. The dielectric properties of water at a frequency of 2.216GHz are reported in Table 8.10; for these calculations, the density of water is assumed to be 1000kg/m^3. The densities of the organic coal components (carbon content, dry, mineral matter free basis) of group 1 and 2 coals were approximated according to their measured volatile matter content (dry mineral matter free basis) and the corresponding density as given in Figure 8.13.

Most of the minerals have similar densities and dielectric properties and, for simplicity, these minerals have been grouped together with an assumed common density of approximately 2700kg/m^3. Exceptions were made for pyrite which has significantly different dielectric properties and a higher density (5000kg/m^3) and kaolin due to the high concentration and marked difference in dielectric properties. Results for both group 1 and 2 coals are shown in Table 8.11.

Table 8.11 Dielectric properties of coal on a dry, mineral matter free basis

Coal	Bulk coal Relative Complex Permittivity $\varepsilon^*=\varepsilon'-j\varepsilon''$ (dimensionless)		Mineral and moisture content based on Normalative analysis (Appendix B) %		Volatile matter (dmmf) %	Organic coal (dry mineral matter free) Relative Complex Permittivity $\varepsilon^*=\varepsilon'-j\varepsilon''$	
	ε_r' 60 ^{o}C	ε_r'' 60 ^{o}C	Mineral content	Moisture		ε_r' 60 ^{o}C	ε_r'' 60 ^{o}C
F-1	2.93	0.1657	2.01	1.6	4.8	2.52	0.1351
F-2	2.49	0.0539	4.55	0.8	13.4	2.43	0.0470
F-3	2.34	0.0818	3.18	1.0	29.8	2.22	0.0725
F-4	2.77	0.2087	6.62	5.8	37.6	1.64	0.1121
F-5	2.32	0.1389	4.28	6.1	38.7	1.21	0.0580
F-6	2.86	0.1564	5.61	6.1	40.4	1.63	0.0702
F-7	2.45	0.1252	6.11	4.7	41.6	1.58	0.0656
F-8	3.51	0.3153	9.59	13.3	45.1	0.74	0.0598
P-1	2.87	0.1848	21.48	3.7	38.0	2.22	0.1349
P-2	1.30	0.0772	15.48	5.2	39.3	0.93	0.0306
P-3	2.84	0.1684	19.60	4.7	39.6	1.79	0.1168
P-4	2.28	0.0658	22.22	1.9	38.3	1.52	0.0239
P-5	2.69	0.1274	17.73	5.0	41.4	2.02	0.0764
P-6	2.44	0.0914	18.78	2.3	36.4	1.67	0.0410
P-7	2.58	0.0906	6.10	3.6	37.7	1.65	0.0410

The results show (Table 8.11) that the relative loss factor (ε_r") for dry, mineral free coal generally increases with increasing coal rank. The relative dielectric constant (as evaluated from the mixture-equation) also increases with coal rank (Figure 8.14). Dielectric constants evaluated from the mixture-equation exhibit a change in trend reported by the measured dielectric constants for these coals (as-received). Both low and high rank coals have relatively higher measured dielectric constants than medium rank coals (Figure 8.14).

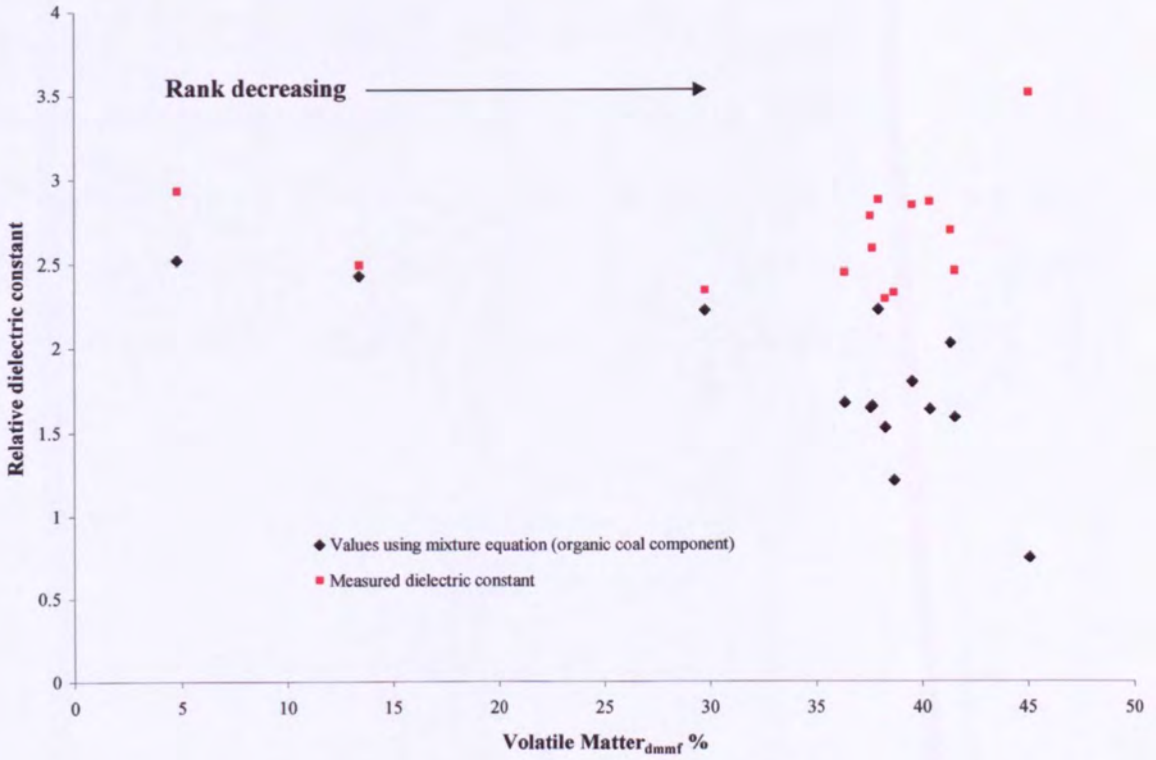


Figure 8.14 Calculated dielectric constants for the organic component of group 1 and 2 coals using the Landau-Lifshitz formula (dry, mineral matter free basis)

Low rank coals generally contain greater quantities of inherent moisture, group 1 and 2 low rank coals also exhibit the highest mineral matter content (up to 20%, in the case of group 2 coals). The increase in moisture and mineral matter in low rank coals increases the dielectric constant value and the evaluated dry, mineral free coal dielectric constants are shown to decrease with decreasing rank. Similar measured results were reported from (moisture-free) group 1 coals at 180°C (Figure 8.7) indicating that the moisture content may be a major contributing factor to measured and calculated (by mixture equation) dielectric constant variation.

8.12 Theoretical heating rate and dependence on electric field strength

Through the manipulation of standard dielectric and physical equations both the theoretical heating rate and the electric field strength can be estimated with a knowledge of a particular material's dielectric and physical properties (Florek 1995). The power absorbed by a material is expressed in (terms of dielectric properties) Equation 2.8, and the power absorbed per unit volume can be evaluated by Equation 8.15.

$$P = 2.\pi.f.\mathrm{E}_{\mathrm{insitu}}{}^{2}.\varepsilon_0.\varepsilon_r'' \qquad (2.8)$$

$$P_a = 2.\pi.f.\mathrm{E}_{\mathrm{insitu}}{}^{2}.\varepsilon_0.\varepsilon_r''.V \qquad (8.15)$$

P Power absorbed (W/m^3)

The energy lost or gained by a material with changes in temperature (heat energy) can be expressed as:

$$Q = m.C_p.\delta\theta \tag{8.16}$$

M	Mass of the body heating (kg)
C_p	Specific heat capacity (J/kg)
θ	Change in temperature (^{0}C)
t	Change in time (s)
Q	Energy absorbed or produced (W)

The power absorbed is therefore ($Q.\delta t$):

$$P_a = \frac{m.C_p.\delta\theta}{\delta t} \tag{8.17}$$

P_a	Power absorbed (W)
M	Mass of the body heating (kg)
C_p	Specific heat capacity (J/kg)
θ	Change in temperature (^{0}C)
t	Change in time (s)

Combining Equation 8.15 and 8.17 gives an expression for the heating rate within a microwave field (Harrison 1997):

$$\frac{\delta\theta}{\delta t} = \frac{2.\pi.f.\mathrm{E}_{\mathrm{insitu}}{}^2.\varepsilon_0.\varepsilon_r''.V}{m.C_p} \tag{8.18}$$

Rearranging Equation 8.18, making the electric field strength inside the material the subject:

$$E_{\text{insitu}} = \sqrt{\frac{\rho . C_p . \delta\theta}{2\pi . f . \varepsilon_o . \varepsilon_r^{"} . \delta t}} \qquad (8.19)$$

However, this assumes no gain or loss of energy from conduction or convection and uniform heating. Previous studies imply that, if a small load is heated within a cavity and the skin depth is high, dielectric heating is the predominant mode of heating and conduction may be negligible (Ishii 1995).

Assuming the density (1000kg/m^3) and specific heat capacity (4200J/kgK) for water do not change significantly over a temperature range 25<T<75°C, and assuming that the loss factor for water between this temperature range equates to (320/T) (T is in Celcius) (Metaxas 1983). Then by measuring the heating rate of water within a microwave cavity, the electric field strength inside the water can be approximated. Equation 8.19 can be summarised as the following assuming no evaporation of water:

$$E_{\text{insitu}} = \sqrt{\frac{1000 \times 4200 . \delta\theta}{2\pi \times 2.45 \times 10^9 . \varepsilon_o . \varepsilon_r^{"} . \delta t}} \qquad (8.19)$$

It is known that the electric field strength may vary within a multi-mode cavity, with the use of a silica glass container, and maintaining the water volume, the cavity electric field strength can be estimated upon measurements of the heating rate and relationships between the electric field strength inside the water and the external field. A grid was marked out on the base of a 0.65kW power microwave cavity (2.45GHz frequency). The heating rate was measured at various positions within the cavity and the average electric field strength was determined for each grid sectional area. Figure 8.15 shows the variation in the heating rate of water within such a cavity.

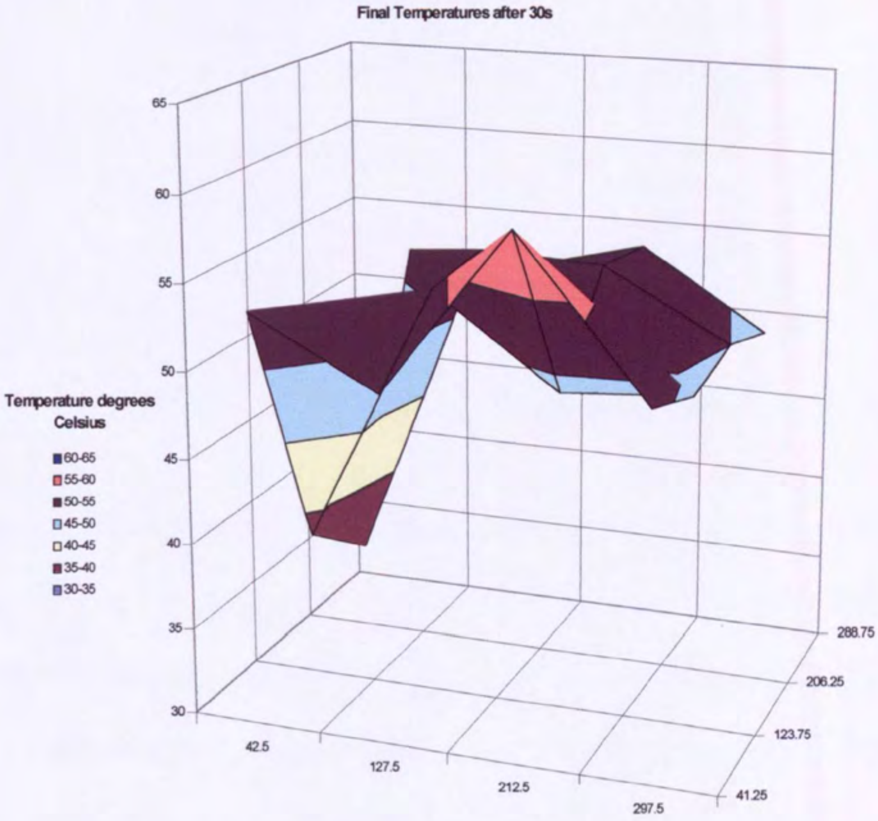


Figure 8.15 Changes in the heating rate of water with position within a 0.65kW multi-mode microwave cavity

The average heating rate of water was 1.1°C/s and the calculated average electric field strength (root mean square basis) inside the water was 1990v/m. Attempts have been made by previous workers (Metaxas 1983) to predict the external electric field strength from a water load (Equation 8.20).

$$E_{ext}^{2} = \frac{Pr}{3c\varepsilon_{o}A\tau'} \qquad (8.20)$$

E_{ext}	Electric field strength of microwave cavity (v/m)
P	Power absorbed (W/m^3)
c	Speed of light (m/s) 3×10^8 m/s
ε_0	Permittivity of free space 8.85×10^{-12} (F/m)
τ'	Constant approximated as 0.25
A	Coefficient of absorption (F/m^2) given by Equation 8.10

$$A = \left(1 - e^{-2r/D_s}\right) \qquad (8.10)$$

r	Radius of particle (m)
D_s	Skin depth (m)
e	Napierian logarithm base approximately equal to 2.7182818285 (10dp)

Following this procedure and applying Equation 8.20, the average cavity electric field strength was 9980v/m. Assuming the cavity electric field strength is constant, theoretical heating rates (within this cavity) for other materials can be derived from a combination of Equation 8.20 (material's internal electric field strength) and Equation 8.18 (heating rate) providing the density, specific heat capacity and particle size are known.

8.12.1 Theoretical heating rates of coal

Table 8.12 Theoretical heating rates of group 1 coal (60°C)

Particle size-0.01m diameter spherical particles						
Coal	**$\varepsilon' \times 10^{-12}$ (F/m)**	**$\varepsilon'' \times 10^{-12}$ (F/m)**	**A (F/m^2)**	**ρ (kg/m^3)**	**E_{insitu} (v/m)**	**$\delta\theta/\delta t$ (°C/s)**
F-1	25.93	1.47	0.049	1530	5920	0.41
F-2	22.04	0.48	0.017	1420	6220	0.16
F-3	20.71	0.72	0.027	1300	6300	0.27
F-4	24.51	1.85	0.062	1300	5986	0.63
F-5	20.53	1.23	0.046	1310	6280	0.46
F-6	25.31	1.38	0.046	1310	5960	0.46
F-7	21.68	1.11	0.040	1320	6210	0.40
F-8	31.06	2.79	0.083	1330	5610	0.81

Densities approximated from Figure 8.13 (Elliot 1981).
Specific heating capacity of coal taken as an average value (1254.5J/kgK), (Clark S P 1966).

The theoretical heating rates of group 1 coals were calculated (Table 8.12) assuming the coal particle shape is spherical and the particle diameter is 0.01m. The densities of all coals were estimated from Figure 8.13. Heating rates may increase in low rank coals due to their increased moisture content.

The theoretical and measured bulk heating rates of microwave treated coals (8 minutes exposure time, 0.65kW, 2.45GHz) are given in Table 8.13, measured heating rates were taken immediately after microwave treatment (Chapter 6.13).

Table 8.13 Theoretical and measured heating rates of microwave heated coal (60°C)

Theoretical heating rate-0.01m diameter spherical particles						Measured heating rate
Coal	**$\varepsilon' \times 10^{-12}$ (F/m)**	**$\varepsilon'' \times 10^{-12}$ (F/m)**	**A (F/m^2)**	**ρ (kg/m^3)**	**$\delta\theta/\delta t$ (°C/s)**	**$\delta\theta/\delta t$ (°C/s)**
P-2	14.20	0.68	0.031	1310	0.31	0.42
P-3	25.11	1.49	0.050	1310	0.49	0.52
P-5	23.83	1.12	0.039	1320	0.38	0.38

Similarities can be drawn between the measured heating rates and the theoretical heating rate, particularly for P-3 and P-5 coals. Some tolerance would be expected, the dielectric properties were measured at 2.216GHz frequency and the sample size and shape differ from assumption made for theoretical calculation (coal pellet samples, cylindrical shape, diameter 0.01m). The measured heating rates were recorded following microwave heating at 2.45GHz from samples of rod mill feed size characteristics (100% passing 0.05m sieve aperture size).

8.12.2 Theoretical heating rates of selected minerals

The theoretical heating rates of selected minerals common to coal are given in Table 8.14 (calculated via Equation 8.20 and Equation 8.18), measured heating rates of these minerals have been included for comparison. Measured heating rates were taken on 50g samples of particle sizes <125μm diameter at a power of 0.65kW and at an operating frequency of 2.45GHz (Harrison 1997).

Table 8.14 Theoretical and measured heating rates of selected minerals (60°C)

Theoretical heating rate-0.01m diameter spherical particles						
	Pyrite	**Quartz**	**Dolomite**	**Kaolin**	**Mica**	**Calcite**
$\varepsilon' \times 10^{-12}$ (F/m)	62.57	16.37	25.58	23.72	25.22	27.08
$\varepsilon'' \times 10^{-12}$ (F/m)	9.40	0.44	0.41	1.45	0.36	0.48
A (F/m^2)	0.185	0.019	0.014	0.050	0.012	0.016
ρ (kg/m^3)	5200	2650	2850	2630	2830_{musc}	2710
C_p (J/kgK)	525	765	840	1048	870	860
E_{insitu} (v/m)	4580	6700	6000	6060	6020	5910
$\delta\theta/\delta t$ (°C/s)	1.11	0.16	0.09	0.30	0.08	0.11
Measured heating rate-100% passing 125μm sieve aperture (Harrison 1997)						
	Pyrite	**Quartz**	**Dolomite**	**Kaolin**	**Mica**	**Calcite**
$\delta\theta/\delta t$ (°C/s)	1.00	0.08	0.12	-	0.08	0.08

Densities taken from (Clark S P 1966), (Mica approximated as Muscovite).
Kaolin density taken from (Roberts et al 1989).
Specific heating capacity taken from (Knache et al 1991).
Specific heating capacity of Mica (Muscovite) taken from (Moses A J 1921).

The data provided illustrates the minerals expected to heat to the greatest extent, pyrite has a heating rate approximately twice that of most coals, kaolin has a similar heating rate whilst all other minerals tested exhibit lower heating rates to organic coals (2.45GHz, 0.65kW).

8.13 Dielectric properties of coal conclusions

Through dielectric measurement of these coals and a selection of associated minerals, the principle heating sources were identified as pyrite and water.

Dielectric properties (dielectric constant and loss factor) are affected by both coal temperature and microwave frequency. The latter exhibits no specific trend, however, the dielectric properties do change but not to any appreciable extent with frequency. Substantial reductions in coal dielectric constant and loss factor values occur between 80-180°C and are attributed to moisture removal. The dielectric properties of coal are dependent on coal rank, both high and low rank as-received coals exhibiting the greatest values of dielectric constant and loss factor. These properties decrease with low rank coals on a dry, mineral matter free basis (calculated dielectric properties from Landau-Liftshifz mixture equation). Hence, the increase in permittivities of low rank as-received coal is due to the increase in moisture of low rank coals. Microwave treated samples result in reductions in the permittivites with increased microwave exposure time, similar reduced values are presented following drying.

The penetration depth of the microwave (which decreases with increase frequency) field ranged from 0.2m to 0.5m depending on coal properties (independent of rank) and substantially increased, particularly for low rank coals on a dry basis.

High and low rank as-received coals exhibit the greatest values of dielectric conductivity and coefficient of absorption, and as such are considered to absorb (as opposed to reflection and transmission) microwave energy to the greatest extent. Absorption capability increases with rank on a dry basis.

From the selection of minerals and measured dielectric properties of water, pyrite and water show substantial absorption capabilities (greater than coal), however, as a result have much shorter penetration depth values. Both water and pyrite heat at twice or more than the heating rate of coal (range between 0.2 to 0.8°C/s). Quartz, mica, dolomite, kaolin and calcite heat at a lower rate (approximately 0.1°C/s) and are expected to transmit microwave radiation.

Dielectric properties provide a basis for preliminary grindability theories (Chapter 6), (expansion of specific minerals and associated stress development from steam production) in so much that steam production would clearly occur following prolonged exposure and coal components would expand at different rates depending upon their heating rates. Results from this investigation show that the heating rates of coal are relatively low within a 0.65kW power input multi-mode cavity. Clearly the heating rate would increase with increasing microwave power input, but the most substantial affect to heating rates may occur at higher cavity electric field strengths.

CHAPTER NINE

MECHANISMS FOR MICROWAVE ENHANCED COAL GRINDABILITY

9.1 Introduction-Mechanisms for microwave enhanced coal grindability

This section discusses the possible mechanisms accounting for the changes in coal grindability upon heating. Analyses of microwave and muffle furnace heating data has shown reductions in coal moisture and volatile matter content after heating (Chapter 6 and 7). In particular, there is evidence to suggest that coals of a high mineral matter and moisture content exhibit the most significant improvement in grindability when heated. Accordingly, it is possible that these two features may control the changes in coal grindability following microwave treatment.

9.2 Mechanisms for improved coal grindability

The bulk temperature of microwave treated coals were measured (Chapter 6.13) at temperatures up to 250°C and an analysis of theoretical heating rates suggest that the temperatures of some inherent minerals (in particular, pyrite) may exceed the coal bulk temperature. As discussed (Chapter 8), the constituents of a coal heat at different rates within a microwave field. They may expand or contract with temperature as each material has a unique expansion coefficient rate with temperature. The volumetric expansion of a solid is related to material specific properties (Coefficient of thermal expansion) and temperature by Equation 9.1.

$$(V_2 - V_1) = \alpha_v . V_1 . (\theta_2 - \theta_1) \tag{9.1}$$

α_v	Coefficient of volumetric expansion (K^{-1})
V_1	Original volume (m^3)
V_2	Final volume (m^3)
θ_1	Original temperature (°C)
θ_2	Final temperature (°C)

Table 9.1 shows the coefficient of volumetric expansion of those minerals generally associated with coal.

Table 9.1 Volumetric expansion of common minerals associated with coal (Clark 1966)

Coefficient of volumetric expansion for selected minerals at increasing temperature (K^{-1})						
Temperature (°C)	**Pyrite ($\times 10^{-6}$)**	**Quartz ($\times 10^{-6}$)**	**Dolomite ($\times 10^{-6}$)**	**Kaolin ($\times 10^{-6}$)**	**Mica ($\times 10^{-6}$)**	**Calcite ($\times 10^{-6}$)**
20	-	34.0	11.4	-	8.5	-
100	21.9	36.2	-	-	8.5	10.5
200	52.9	78.1	-	-	-	28.5
400	129.1	189.4	-	-	-	76.5
600	-	452.3	-	-	-	139.5
800	-	442.4	-	-	-	-

Data for Dolomite-Handbook of Physical Quantities (Meilikhov 1997)
Data for Mica-Smithsonian Physical Tables (Forsythe 1954)

Pyrite and calcite decompose at high temperatures (e.g. pyrite oxidises at approximately 300°C). Generally, the expansion coefficient increases (expansion rate) with increasing temperature. This can also be observed with quartz up to 600°C. Quartz undergoes a phase conversion at 573°C, the transition between α and β quartz accounting for the significant increase in coefficient of expansion followed by the decrease in coefficient of expansion at higher temperatures. Furthermore, the coefficient of volumetric expansion for water at constant pressure increases (from 45.6×10^{-8} for ice at -50°C, 182×10^{-6} for water at 20°C and 4187×10^{-6} for steam at 120°C, Meilikhov 1997, Forsythe 1954) to a greater extent than most mineral inclusions over the temperature range 20°C-120°C.

The reductions in total moisture and volatile matter content (Chapter 6) of treated coals suggest that there is steam generation and volatile gas production with increasing temperature. There may be associated changes in coal structure and internal pressures may be generated as a result of gas and steam evolution from within the structure. In particular, once a gas is produced, it can be assumed that internal stresses would increase from increasing gas pressure with temperature. If the gas is confined i.e. no volume change, and assuming ideal gas laws (Equation 9.2), as temperature increases the pressure increases linearly. The pressure will continue to increase until either the heating ceases or the volume increases. Internal structural stress would increase as a result of the increasing pressure and may initiate crack propagation.

$$P_g \mathrm{V}_g = n\mathrm{R}T_g \tag{9.2}$$

P_g	Pressure of gas ($\mathrm{N/m^2}$)
$\mathrm{V_g}$	Volume of gas ($\mathrm{m^3}$)
T_g	Temperature of gas (K)
n	Number of moles in fluid (kmol)
R	Universal gas constant (8.314kJ/kmol K)

There is evidence to show cracks are produced when coal is heated, possibly as a result of internal stresses generated by the volumetric expansion of both solid and gas components in the coal.

Previous results (Chapter 6) have shown improvements in coal grindability with heat treatment, volumetric expansion would also produce internal stresses as the coal temperature is reduced. To demonstrate this, two (500g) representative P-7 coal samples were prepared for cryogenic treatment, one of the samples was enclosed within an insulation flask surrounded by ice at a temperature of –1°C. The second sample was enclosed within an insulation flask and covered with liquid nitrogen (-196°C), the samples were removed after 1 hour of exposure. The two samples and a further non-treated sample (reference) were milled (Chapter 6.8, 100rpm speed of rotation, 7 rods used as grinding media) and size distribution determined after 5, 20 and 45 minutes of milling.

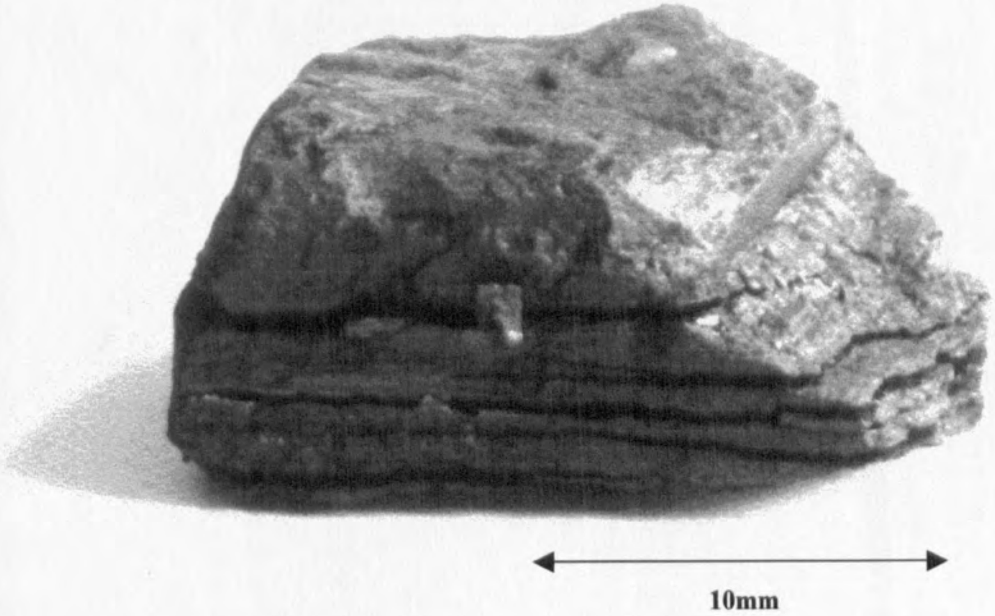


Figure 9.1 Visible crack formation following microwave heating

Table 9.2 Effect of reduced temperatures on coal grindability

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
Reference (20°C)	100	100	100
As-received (-1°C)	89	85	99
As-received (~ -200°C)	71	62	90

Results show (Table 9.2) no change in coal grindability; after 45 minutes of milling, for the sample exposed to –1°C. However, the sample covered in liquid nitrogen showed a 10% reduction in RWI after 45 minutes of milling, possibly because of the significant temperature reduction and the subsequent effect of the differences in contraction between the minerals, water and coal. The expansion rates for most minerals associated with coal and water increase with temperature. The coefficient of expansion for ice is significantly lower than that of water. Hence, the extent of the effect upon grindability may not be as significant as that produced from heat treatment.

The behaviour of the material to which the stress is applied will alter according to the chemical and physical properties of the material (composition, size and location of composites i.e. mineral matter content and inherent moisture content). If the force is sufficiently rapid, intensive or extensive duration, some breakage of the structure will result (Hookes law). The in-situ method of heating from microwave radiation may promote an increased rate of applied force due to increased heating rates of some inherent composites. Expansion forces may be low and insignificant, not sufficient to deform the bulk of the structure. However, they may be sufficient to extend existing imperfections or discontinuities in the material i.e. cracks and pores, which are considered points of weakness or stress concentration points (Brown 1958).

9.2.1 Points of structural weakness

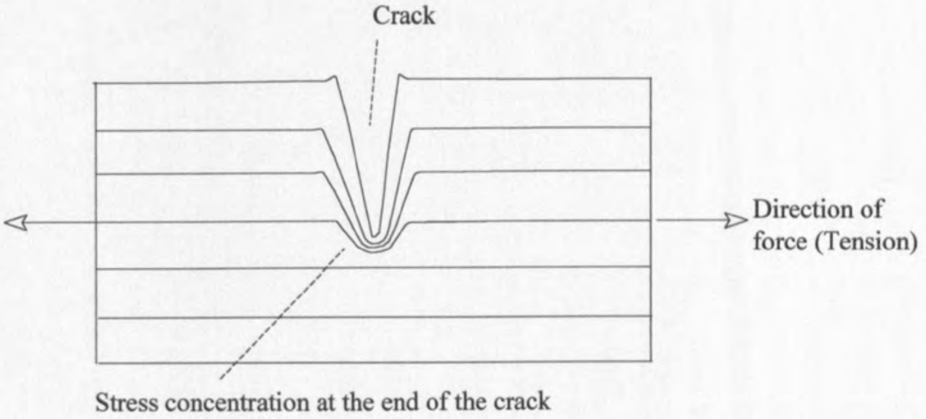


Figure 9.2 Effect of crack length and radius of curvature on stress concentration

Stress concentrates at non-uniform structural points. Any cracks, pores, joints and boundaries between mixtures may act as stress concentration points. Considering an existing crack within a uniform body, the maximum stress acts at the point of maximum concentration i.e. the crack tip (Figure 9.2). Assuming the crack is elliptical in shape the maximum stress can be related to the crack dimensions (Equation 9.3, Lowrison 1974).

$$\text{Crack stress} = \text{Average stress} \times \left(1 + 2\sqrt{\frac{\text{length of major axis of crack}}{2 \times \text{radius of curvature at tip of crack}}}\right) \qquad (9.3)$$

The stress increases as the length of the crack increases, additionally, as the length increases the radius of curvature decreases. Once the stress is sufficient to initiate crack elongation (crack tip stress = crack initiation stress) and provided that the stress is then maintained throughout the body, the crack will be self-propagating. However, a particular crack requires a minimum energy for elongation initiation. This may be measured assuming the crack initiation stress is related to the potential energy of the crack (Equation 9.4, Lowrison 1974).

$$\text{Crack initiation stress} = \sqrt{\frac{2.G.\gamma}{l}} \qquad (9.4)$$

G	Young's modulus (N/m^2)
l	length of crack (m)
γ	Surface energy per unit surface area (J/m^2)

The crack initiation is related to the crack length, if the crack length is very small then the crack stress initiation can be larger than the stress required to deform the bulk material (phase change as an extreme). The minimum crack length is considered to be that to which Van Der Waal's forces no longer effectively act (Lowrison 1974). Hence, cracks or pores below the minimum length are not generally considered points of weakness.

In natural heterogeneous materials, the stress throughout the body is non-uniform and cracks may propagate and cease before catastrophic structural failure (breakage) as the crack interacts with other cracks, pores or inclusions that act as 'crack stoppers' (Brown 1958).

Cracks and pores are considered the major source of material fracture points and as such their importance is unquestionable. Particle size is considered an influencing factor on grindability, smaller particles may not produce cracks to the same extent as larger particles and as such resist grinding more effectively i.e. the smaller the particle the greater the resistance to grindability (Lowrison 1974).

To this end, grindability will be improved providing the heat treatment supplies sufficient energy (to the rate and intensity of the force from expansion) to propagate crack formation. The location, size and quantity of such cracks and their propagation depend upon the inherent constituents and there affect as a consequence to heat treatment and volumetric expansion.

9.3 Improved microwave grindability

Results (Chapter 6 and 7) indicate that microwave enhanced grindability improves with decreasing coal rank and increasing mineral matter content. The most significant improvements in coal grindability have been measured on low rank coal (Chapter 7), which may result from the high inherent moisture content of such coals. The question may be asked: could the grindability be improved further by increasing the inherent moisture content of a coal? Clearly it would be impractical to increase the mineral matter content of a coal as the benefits from heat treatment require the minerals to be enclosed within or attached to coal particles.

9.3.1 Potential to increase inherent moisture content

All coals contain both surface moisture and inherent moisture. The surface moisture would originate from exposure to the atmosphere (Relative Humidity); this type of moisture would absorb microwave radiation, rapidly heat, change phase and evaporate from the surface with presumably little effect on the coal structure. Inherent moisture originates from the coalification process and as such is part of the internal structure. This form of moisture would also rapidly heat, however, due to confinement may change phase under increased pressures that may propagate micro-cracks. Coals are naturally porous (Chapter 3.3), it is possible that inherent moisture may be located within or near to existing pores or micro cracks. The average pore-opening diameter of coal is less than 200Å (0.2μm)(Figure 9.3). Hence it is expected that any inherent moisture within the pores would significantly weaken the structure.

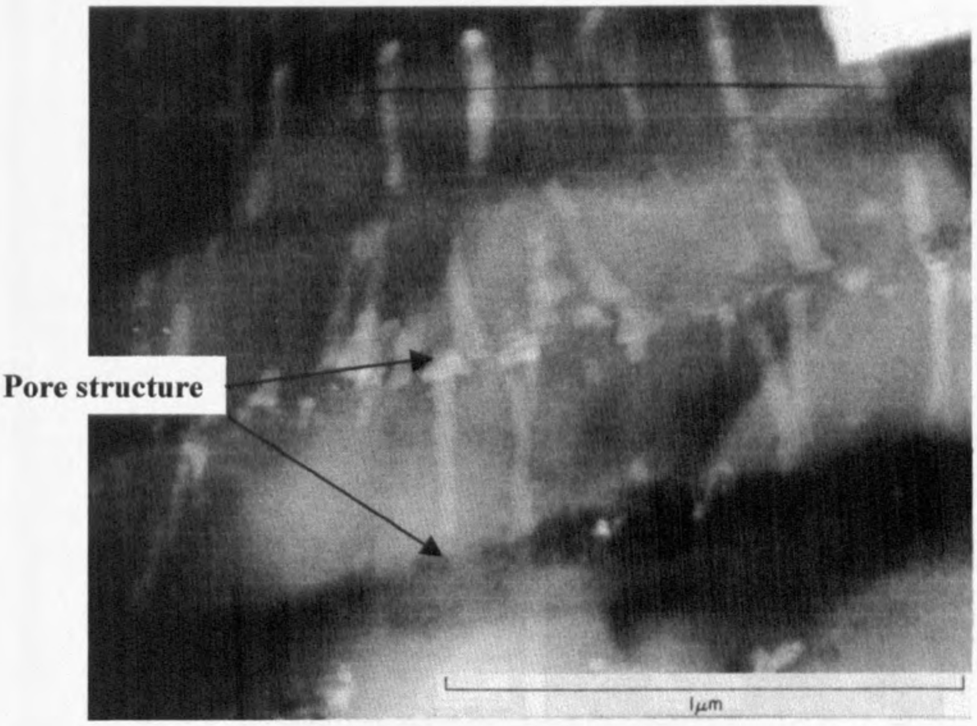


Figure 9.3 Scanning Electron Micrograph image of coal pores (Harris 1976)

However, it is believed that, increasing microwave grindability benefits from an increased moisture content would be impractical with simple additions of water under atmospheric conditions as the pore opening would be too small to allow water penetration.

9.3.2 Microwave grindability of P-2 and P-7 coals with increased moisture content

Representative (500g) samples of P-2 and P-7 coals were prepared in a similar method to that described in Chapter 7.5, size distributions range between -50mm +500μm. Both coals were of medium rank, P-2 is a typical power station feed coal whilst P-7 was chosen because of its low ash content which may affect heat treatment results. The coals were exposed to ambient atmosphere for twenty four hours before being enclosed within air-tight containers to retain their constant relative humidity equilibrium.

Two of the samples from each coal were submerged in 400ml of distilled water for 48 hours, the samples were irradiated following excess water drainage. The ensuing microwave treatment was constant for all coals (samples exposed to microwave radiation for 8 minutes at 0.65kW power input and at 2.45GHz frequency). Microwave conditions were selected to produce reasonable grindability improvements for medium rank coals (Chapter 6.11).

A sample from each coal tested was heated within a muffle furnace maintained at 110°C for 180 minutes to ascertain the effect of drying upon coal grindability. A further sample from each coal; was treated by both the furnace and by microwave heating methods to compare grindability effects after subsequent milling tests (comparison between furnace and microwave treatment).

Following heat treatment, representative sub-samples were taken for proximate analysis. The quantity of coal taken for proximate analysis depended upon that remaining after heat treatment, 450g of each coal remained for grindability tests. Subsequent laboratory rod milling (Chapter 6.10) was carried out to determine any improvements in grindability. Results are expressed in terms of the Relative Work Index.

Table 9.3 Relative Work Index of P-2 coal after microwave and furnace treatments

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
Reference	100	100	100
Furnace	82	79	58
Furnace + Microwave	78	67	47
Microwave	81	85	67
H_2O_l + Microwave N°1	78	79	51
H_2O_l + Microwave N°2	81	92	73

Results for tests carried out with P-2 coal (Table 9.3), show that furnace treatment can produce greater improvements in grindability with comparison to microwave treatment. Combining furnace and microwave heating produced the greatest reduction in Relative Work Index (greatest improvement in grindability). Repeatable results were not obtained with coals that had been subjected to water additions. Proximate analysis results (Table 9.4) may be used to rationalise these inconsistencies. The moisture content for all treated coals was reduced, the inconsistent values of RWI may be a result of the variation in mineral matter content between samples. The coals with the greatest reductions in Relative Work Index also correspond to the greatest ash content. As there are significant variations in each coal sample ash content, comparisons between heating methods and increased moisture contents are difficult to evaluate from these results.

Table 9.4 Proximate analysis (as-received basis unless stated) of P-2 coal after microwave and furnace treatments

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Reference	4.49	12.37	31.55	51.59	37.95
Furnace	1.84	17.47	28.86	51.84	35.76
Furnace + Microwave	1.31	13.51	29.48	55.70	34.61
Microwave	2.26	12.31	30.51	54.93	35.71
H_2O_l + Microwave N°1	2.48	15.50	29.31	52.71	35.74
H_2O_l + Microwave N°2	2.93	13.52	31.26	52.28	37.42

P-7 coal was chosen due to the reasonably low ash mineral content (less than 5%). Results (Table 9.5) following heat treatment and milling show improvements in grindability i.e. reductions in RWI. However, the RWI values are uniform irrespective of water additions (85% after 45 minutes of milling) and repeatable results could be obtained. This suggests that additions of water have had no effect on the grindability, any increase in total moisture prior to microwave treatment represents surface moisture only (moisture content of 8.2% was measured for samples subjected to water addition prior to microwave treatment).

Table 9.5 Relative Work Index of P-7 coal after microwave and furnace treatments

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
Reference	100	100	100
Furnace	77	68	90
Furnace + Microwave	80	64	84
Microwave	73	67	84
H_2O_l + Microwave N°1	77	68	85
H_2O_l + Microwave N°2	75	69	85

Surface liquid moisture is believed to have no penetration capabilities into the coal structure and as such no effect on grinding resistance after steam generation. The 5% difference in RWI between microwave treated coals and furnace treated coal may result from the increase in temperature and subsequent effect on the volumetric expansion between heating methods. The furnace was maintained at a temperature of 110°C and as such, there is expected to be some effect by the volumetric expansion of coal mineral constituents on coal grindability. However, the bulk temperatures during microwave treated coals reached a temperature of 180°C (K-type thermocouple measurement) which may have a greater effect on volumetric expansion. Additionally, proximate analysis (Table 9.6) shows that the total moisture content of microwave treated samples are reduced further than the furnace treated coal, which may account for the difference in RWI via steam generation.

Table 9.6 Proximate analysis (as-received basis unless stated) of P-7 coal after microwave and furnace treatments

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Reference	3.71	4.46	35.54	56.30	36.56
Furnace	2.60	4.64	33.03	59.73	35.61
Furnace + Microwave	1.41	4.57	34.59	59.43	37.15
Microwave	1.86	4.96	34.21	58.98	36.71
H_2O_l + Microwave N°1	2.26	4.24	34.20	59.29	36.58
H_2O_l + Microwave N°2	1.75	5.72	32.96	59.57	35.62

9.3.3 Steam liberation restriction

Similar tests were carried out (P-7 coal only) to ascertain whether the use of surface agents could be used to prevent or slow inherent steam liberation. Coal samples were prepared as described in Section 9.3.2, samples were submerged in 400ml distilled water-surfactant (50g) solution (Calgon – brand name, 15% polycarboxylates, 30% Zeolites, typical detergents contain ~15% surfactants). Microwave irradiation commenced following excess solution drainage. Microwave and milling conditions were as described previously (Section 9.3.2).

Table 9.7 Relative Work Index of P-7 coal with surfactant pretreatment addition after microwave exposure (8 minutes, 0.65kW at a frequency of 2.45GHz)

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
Reference	100	100	100
H_2O + Surfactant +Microwave N°1	71	62	84
H_2O + Surfactant +Microwave N°2	71	62	83

Results (Table 9.7) are similar to those obtained in Section 9.3.2 indicating no effect by the surfactant upon microwave grindability. Proximate analysis (Table 9.8) shows similar reductions in moisture content as reported for P-7 coal exposed to microwave radiation irrespective of water/surfactant addition, indicating that the surfactant did not restrict or enhance steam liberation.

Table 9.8 Proximate analysis (as-received basis unless stated) of P-7 coal with surfactant pretreatment addition after microwave exposure (8 minutes, 0.65kW at a frequency of 2.45GHz)

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Reference	3.71	4.46	35.54	56.30	36.56
H_2O + Surfactant +Microwave N°1	1.99	4.03	32.65	61.33	34.74
H_2O + Surfactant +Microwave N°2	1.74	6.55	32.96	58.75	35.94

9.3.4 Enhanced microwave grindability of P-7 coal with the addition of steam to increase the inherent moisture content.

Tests were carried out (P-7 coal) to determine whether the inherent moisture content could be increased with the addition of steam. Samples were prepared as described in Section 9.3.2. Samples were contained and exposed to a constant flow of steam for 1 hour. A sample was milled (Chapter 6.11) immediately after steam addition and two further steam treated samples (to demonstrate repeatability) were exposed to microwave radiation (8 minutes, 0.65kW, 2.45GHz) prior to milling. Size distribution data following 5, 20 and 45 minutes milling is expressed in terms of the RWI (Table 9.9).

Table 9.9 Relative Work Index of P-7 coal following the addition of steam and microwave treatment

Coal	Relative Work Index %		
	Grinding time		
	5 min	20 min	45 min
Reference	100	100	100
As-received+H_2O_G	71	62	95
Microwave + H_2O_G N°1	71	58	84
Microwave + H_2O_G N°2	71	58	84

Microwave treated samples (Table 9.9) show a similar size distribution, hence similar RWI as previous microwave treated P-7 coal samples; indicating that steam addition had no further effect on coal grindability. Results from the as-received, steam treated sample show little reduction in RWI after 45 minutes of milling. This may be the result of the increase in bulk temperature and associated expansion of mineral inclusions rather than any increase in the inherent moisture content. Proximate analysis results (Table 9.10) show no increase in the as-received steam treated sample suggesting the moisture content could not be increased by means of steam addition. Microwave treated samples show similar reductions in total moisture content as previously reported (Section 9.3.2).

Table 9.10 Proximate analysis (as-received basis unless stated) of P-7 coal with the addition of steam before and after microwave exposure (8 minutes, 0.65kW at a frequency of 2.45GHz)

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Reference (20°C)	3.71	4.46	35.54	56.30	36.56
As-received+H_2O_G	3.62	3.66	35.02	57.70	37.77
Microwave + H_2O_G N°1	2.44	3.75	33.82	59.99	36.05
Microwave + H_2O_G N°2	2.02	3.92	33.94	60.10	36.09

9.4 Conclusions-Mechanisms for microwave enhanced coal grindability

The expansion of inherent minerals and moisture are believed to promote fracture propagation upon coal heating. Results (Chapter 6, 7 and 9) have shown that both microwave and convective heating may improve grindability as a result of different thermal expansion rates of coal inclusions. Microwave heating can induce a weakening of the coal structure to a greater and a lesser extent to that of convective heating depending upon the mineral composition and associated heating rates. Microwave irradiation may promote high heating rates from within the coal structure that is not dependent upon conduction. The extent of the reduction to grinding resistance depends upon the quantity, grain size, proximity and heating rate of the inclusion. Hence, high heating rates and increasing quantities of mineral matter and moisture content may promote the most significant change to coal grindability. Clearly it is not feasible to increase the inherent mineral matter content. Furthermore, results (Chapter 9) suggest that it is impractical to increase the inherent moisture content. Accordingly, when employing microwave radiation as a method of pretreatment, the choice of coal is vital in achieving significant improvements in grindability. Further investigation would be necessary to determine the specific minerals that promote the greatest structural defects and their optimum grain size with relation to coal particle size and grindability.

CHAPTER TEN

PILOT-SCALE CONSIDERATIONS

10.1 Introduction-Pilot-scale considerations

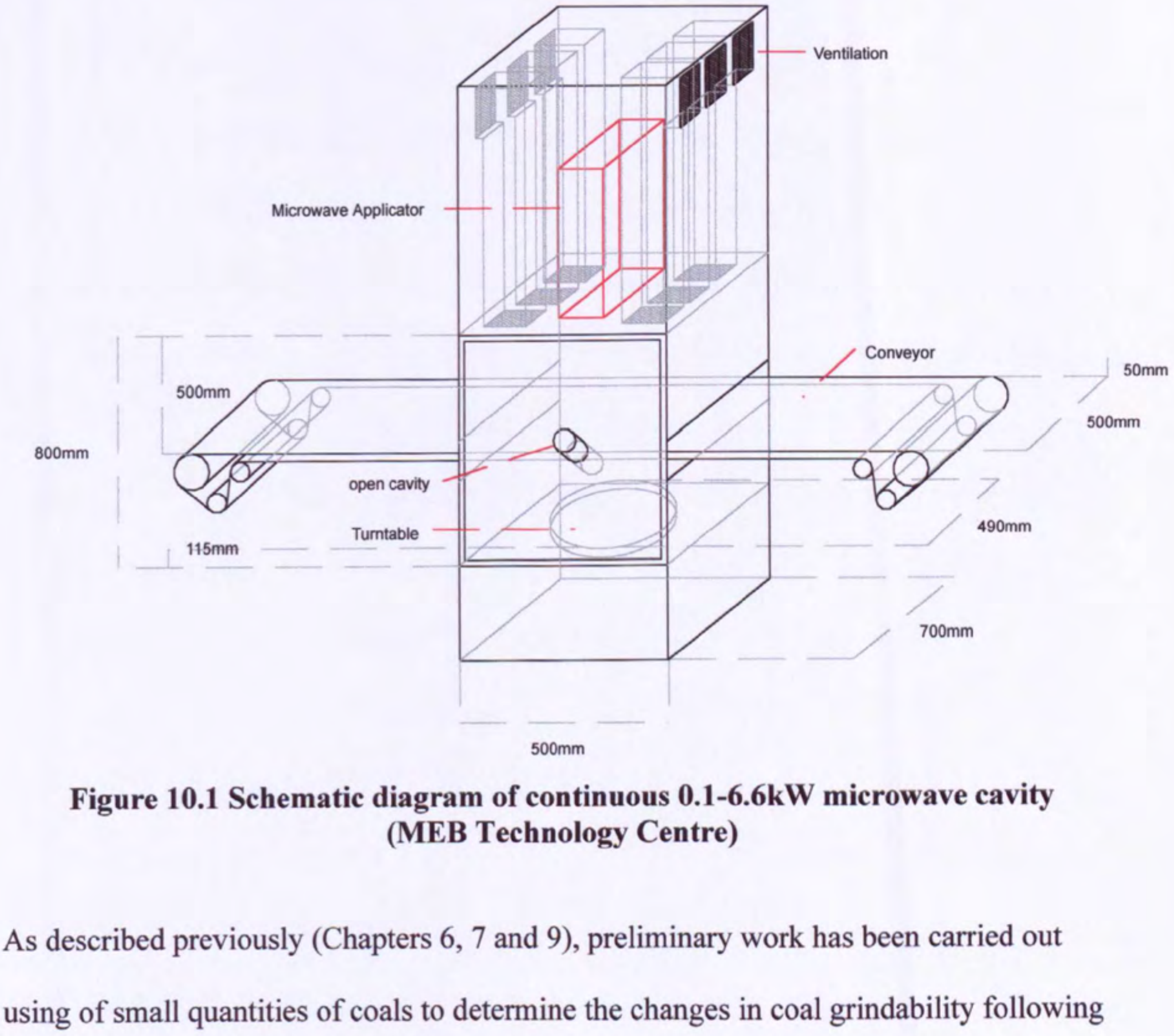


Figure 10.1 Schematic diagram of continuous 0.1-6.6kW microwave cavity (MEB Technology Centre)

As described previously (Chapters 6, 7 and 9), preliminary work has been carried out using of small quantities of coals to determine the changes in coal grindability following microwave treatment. Pilot-scale trials have also been conducted to demonstrate that sizeable quantities of coal could be irradiated and to (i) identify any scale-up problems and (ii) provide a basis for a feasibility analysis.

To this end, a microwave cavity capable of operating at both batch and continuous process modes and at power levels between 0.1kW-6.6kW, was used for pilot-scale trials (Figure 10.1). The operating frequency of the cavity was fixed at 2.45GHz.

10.2 Process considerations

The basis of the microwave pilot-scale trials was to demonstrate the potential applications within the coal industry for microwave technology. Industrial PF production for power generating plant would require a continuous microwave process between feed storage and mills, prior to boiler entry. Such a process would maintain a constant throughput (without sustained coal ignition) and produce uniform heating characteristics and associated improved grindability. Any gases released as a by-product of microwave treatment would be removed for cleaning. A batch process may prove impractical as industrial PF production for power generation relatively high feed rates of coal; this feature may limit batch cavity dimensions, in principle, high loads may result in non-uniform heating (localised heating) and difficulties with handling and storage.

To provide flexibility, the current cavity allows for both continuous (conveyor belt) operation and batch (turntable operation) processing. Cavity ventilation could remove all emissions and reduce hot-air congregation.

10.3 Microwave treatment conditions

The purpose of the current study was to demonstrate that preliminary work can be carried out on sizeable quantities of coal and to assess the variables which need to be considered for industrial-scale microwave treatment. The pilot-scale tests were conducted in two stages, the first being concerned with investigating the practical range of experimental parameters and operating conditions and the second stage with a study of the effect of microwave radiation on an appropriate quantity (0.5 tonne) of coal. Cavity optimisation was primarily concerned with defining the most practical balance between coal quantity, power input and microwave exposure time for the energy required for maximum grindability improvement. Cavity conditions were based on the use of a batch process and it was assumed that a continuous feed could be represented as a succession of batches.

10.3.1 Microwave energy requirements

Each microwave cavity has a unique electric field strength distribution and, in particular, the cavity dimensions (shape) will affect the power absorption efficiency. Accordingly, energy requirements can be expected to differ between cavities for a given reduction in RWI (Relative Work Index) of a coal. Furthermore, coals differ in their composition and structure and each coal will have a different energy requirement for achieving maximum reduction in RWI (Chapter 6.11). Preliminary work (Chapter 6) has also demonstrated that the coal feed size distribution (which clearly would not be uniform for a continuous process) would affect the grindability change arising from microwave treatment. For these reasons, microwave energy per unit weight of coal was not optimised, but energy input was more than sufficient to show considerable improvement in coal grindability.

The final bulk temperature of the coal has been shown to be an indication of the maximum improvement in grindability (Chapter 6.13) and preliminary studies have shown that the maximum reduction in RWI occurred at bulk coal temperatures within the range of 160°C-200°C. A group of samples were irradiated at various power input levels and sample loads. The bulk temperature of the coal (following microwave treatment) was measured using a K-type thermocouple (temperature range –50°C-900°C). For all coals tested (P-5, P-6 and P-8), a power input of approximately 220kWh/t was required to produce temperatures consistently ranging from 160°C-200°C, independent of sample load and input power. On this basis, all subsequent tests were carried out at constant energy input (220kWh/t).

10.3.2 Variation of grindability with increased power input at constant load

Batch (1kg) samples of P-5 coal were exposed to microwave radiation to investigate the effect of increased microwave power input on coal grindability. All samples were of uniform feed size distribution and exposed at constant energy per unit weight (220kWh/t). The microwave power input was set at levels 1.1, 3.3 and 6.6kW corresponding to exposure times of 8, 4 and 2 minutes respectively. Sub-samples (500g) from the three microwave-treated samples and a non-treated sample were prepared and subjected to RWI grindability tests (Chapter 6.11).

Results (Table 10.1) show that the RWI decreased by 30-40% (45 minutes of milling) following microwave treatment. Furthermore, grindability improved with increasing input power from 1.1kW to 3.3kW with similar effects exhibited for the 3.3kW and

6.6kW treated coals. There was no evidence of coal ignition occurred during these pilot-scale tests.

Proximate analyses (Table 10.2) show significant (and similar) reduction in the moisture content of all microwave-treated coals; minor reductions in volatile matter (which reduce with increasing microwave power input) are also noted.

Table 10.1 Relationship between Relative Work Index and microwave input power at constant energy per unit weight (1kg P-5 coal samples, at 2.45GHz frequency)

Coal	Power input (kW)	Relative Work Index %, ground for:		
		(5 min)	(20 min)	(45 min)
Non-treated	-	100	100	100
Exposure 8 min	1.1	79.1	79.3	67.1
Exposure 4 min	3.3	72.2	75.7	59.0
Exposure 2 min	6.6	69.4	74.1	59.0

Table 10.2 Proximate analysis (as-received basis unless stated) of P-5 coals exposed to microwave radiation at constant wieght (1kg)

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Non-treated	3.41	13.48	34.18	48.93	41.13
Exposure 1.1kW	1.65	11.72	36.33	49.80	42.51
Exposure 3.3kW	1.80	14.14	33.55	50.51	39.92
Exposure 6.6kW	1.49	11.26	32.62	54.63	37.39

10.3.3 Grindability variation with increased load at constant power input

A similar set of (P-5) coals of different quantities (1,2 and 3 kg loads) were irradiated at constant microwave power input (3.3kW) for 4, 8 and 16 minutes respectively (constant energy per unit weight) to determine if batch size affects microwave enhanced-grindability. A sub-sample (500g rod mill feed of uniform size distribution) of each coal was taken and the RWI determined (Chapter 6.11).

Results (Table 10.3) show that following 45 minutes milling, the RWI reduces with decreasing coal quantity. As the energy and power input are constant, the major dependant parameter is the sample volume. In particular, this implies that increased sample volumes may be approaching the matched load for the cavity; additionally the coal sample height increases with increased load (sample area maintained), and accordingly, penetration depth may become a factor influencing grindability at increased volumes.

Table 10.3 Relationship between Relative Work Index and coal quantity at constant microwave input power (3.3kW) and at constant energy per unit weight (P-5 coal, 2.45GHz frequency)

Coal	Sample load (kg)	Relative Work Index %, ground for:		
		(5 min)	(20 min)	(45 min)
Non-treated	1	100	100	100
Exposure 4 min	1	72.2	75.7	59.0
Exposure 8 min	2	75.1	77.7	62.6
Exposure 12 min	3	80.5	81.9	65.0

Table 10.4 Proximate analysis (as-received basis unless stated)of P-5 coals exposed at constant microwave power input (3.3kW) and constant energy per unit weight (220kWh/t)

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Non-treated	3.41	13.48	34.18	48.93	41.13
1kg	1.80	14.14	33.55	50.51	39.92
2kg	1.56	11.22	33.47	53.75	38.38
3kg	1.96	10.29	34.03	53.71	38.79

Proximate analyses (Table 10.4) show significant (and similar) reduction in the moisture content of all microwave treated coals; small reductions in volatile matter are also exhibited.

10.3.4 Operating conditions used in scale-up tests

At a constant energy per unit weight of 220kWh/t, results (Tables 10.1 and 10.2) suggest that the input power could be operated above 3.3kW with little variation and imply that low coal bed heights may result in the greatest reductions to RWI. Clearly, during continuous operation a short residence time is preferable and accordingly the maximum power input (6.6kW) is most practical. However, conveyor speed is a limiting factor for this particular design of this machine. The conveyor was currently capable of sustaining speeds ranging from 0.0011m/s (minimum) to 0.25m/s (maximum). For this reason, the feed rate of coal was maintained at 30kg/hour corresponding to a conveyor belt speed of 0.002m/s at an input power of 6.6kW. A maximum of 3kg coal was presented to the microwave field at any given time and the bed height maintained below 50mm (top size of the coal), corresponding to the width of the belt.

10.4 Coal selection for pilot-scale study

Three power station coals of sub-bituminous rank (P-5, P-6 and P-8) were selected for pilot-scale tests. P-5 coal was used exclusively in preliminary tests relating to cavity operating conditions (Section 10.3). P-5 coal was chosen because of its similar Hardgrove Grindability Index (58) to those of the other two coals and because it had a similar pyritic sulphur content to that of P-8 coal (0.52%). Preliminary studies (Chapters 6-9) indicated that inherent moisture and high mineral matter contents may promote RWI reduction following microwave exposure. Proximate analyses of these coals are given in Table 10.5. All coals had a total moisture content greater than 3% and mineral matter contents within the range 12.9% and 21.9%.

Table 10.5 Proximate analysis (as-received basis unless stated) of reference coals

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
P-5	3.41	13.48	34.18	48.93	41.13
P-6	4.41	12.91	30.33	52.35	36.68
P-8	3.17	21.87	26.72	48.24	35.65

Approximately 200kg of P-6 coal was prepared for microwave irradiation and a further quantity was prepared for grinding within a suitably sized pin mill. The coal was pre-crushed (100% passing 3mm screen aperture size) to conform to mill feeder tolerance limits.

The P-8 coal (100% passing 50mm screen aperture size) was of power station mill feed size characteristics prior to comminution (Chapter 3.9) and as such, an appropriately sized mill was chosen for size reduction. Approximately 500kg of P-8 coal was fed into a pilot-scale continuously fed Autogenous Barmac™ Rock-On-Rock VSI crusher operating at speeds of 3000rpm and 5000rpm. A similar quantity was retained for pilot-scale microwave treatment.

10.4.1 Laboratory-scale (P-6, P-8) microwave treated coal grindability tests

Prior to pilot-scale tests, samples of P-6 coal were exposed to microwave radiation to ascertain the extent of the reduction in RWI. The energy per unit weight was equivalent to that for pilot-scale operating conditions. Whilst the laboratory and pilot-scale tests were not comparable (because of differences in cavity design) consideration was given to the extent of the treatment upon grindability (degree of reduction in RWI after laboratory milling).

Two (P-6) coal samples-for laboratory scale (500g) microwave tests-were prepared using cone, quartering and riffling methods. Each coal was subjected to milling within a laboratory-sized rod mill (Chapter 6.8) and size distributions were measured following 5, 20 and 45 minutes milling. A sample was irradiated at an input power of 0.65kW (2.45GHz frequency) for 609s (equivalent to 220kWh/t) prior to milling. The RWI of the microwave treated coal is given in Table 10.6.

Table 10.6 Relative Work Index of –3mm P-6 coal following microwave treatment (0.65kW, 220kWh/t, 2.45GHz, 500g)

Coal	Exposure Time (s)	Relative Work Index %, ground for:		
		(5 min)	(20 min)	(45 min)
Non-treated	-	100	100	100
Exposure 0.65kW	609	86.5	78.0	88.7

Following 45 minutes milling, only a small reduction in RWI was achieved as a result of microwave treatment.

Similarly, samples of P-8 coal were prepared and subjected to microwave exposure in two separate cavities; input powers of 0.65kW and 2.6kW were used at exposure times of 609s and 152s respectively (220kWh/t at 2.45Ghz frequency). Following microwave treatment and 45 minutes milling, results (Table 10.7) show reductions of up to 80% RWI. Furthermore, both microwave treated coals gave similar product size distributions

(Figure 10.2) implying that, for this particular coal, the increase in power input and cavity differences were not issues affecting grindability.

Table 10.7 Relative Work Index of P-8 coal following microwave treatment (2.6kW, 220kWh/t, 2.45GHz, 500g)

Coal	Exposure Time (s)	Relative Work Index %, ground for:		
		(5 min)	(20 min)	(45 min)
Non-treated	-	100	100	100
Exposure 0.65kW	609	8.5	14.3	19.0
Exposure 2.6kW	152	8.3	14.4	19.0

Proximate analyses (Table 10.8) of the exposed (P-8) coals showed substantial reductions in the moisture content. Volatile matter content for both microwave treated coals appeared unchanged.

Table 10.8 Proximate analysis (as-received basis unless stated) of P-8 coals exposed to microwave radiation

Coal	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
Non-treated	3.17	21.87	26.72	48.24	35.65
Exposure 0.65kW	0.48	22.57	27.14	49.82	35.26
Exposure 2.6kW	0.40	24.43	26.45	48.72	35.19

Despite the fact that the microwave treated coals were not milled to PF size, it is inferred (Tables 9.6 and 9.7) that P-8 coal would show the greater benefit from pilot-scale microwave exposure.

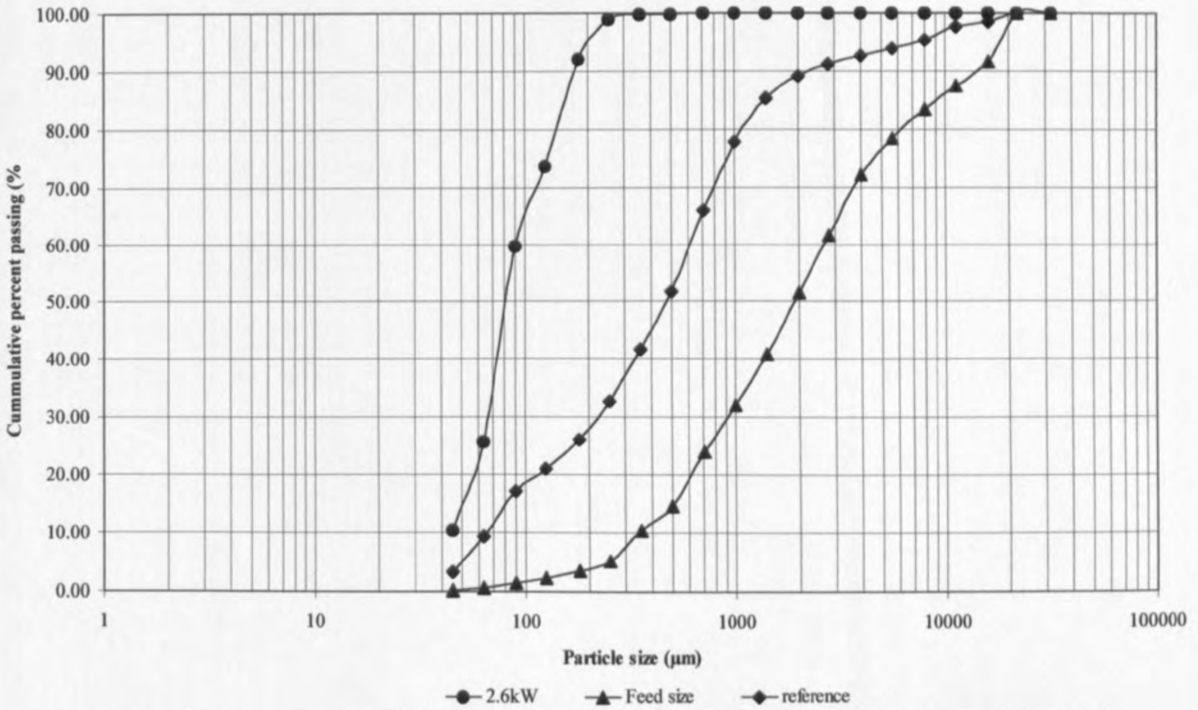


Figure 10.2 Size distributions of microwave treated (2.6kW, 2.45GHz, 220kWh/t) and as-received P-8 coal following 45 minutes milling (500g batch mill load)

10.5 Microwave affect on grindability (pilot-scale)

Following microwave treatment, 500kg of P-8 coal was fed into a pilot-scale continuously-fed Autogenous Barmac^{TM} Rock-On-Rock VSI crusher operating at speeds of 3000rpm and 5000rpm. Samples for size distribution analyses were taken after an appropriate time had elapsed (5 minutes) to allow the machine to reach milling equilibrium. Neither the microwave-treated or as-received samples at either operating speed gave products of PF size characteristics. However, results using operating speeds of 3000rpm and 5000rpm (Figures 9.3 and 9.4) show some preferential breakage (particularly at higher rotor speeds, Figure 10.4) between 5 to 0.5mm for the microwave treated coal, with a reduction in fines production.

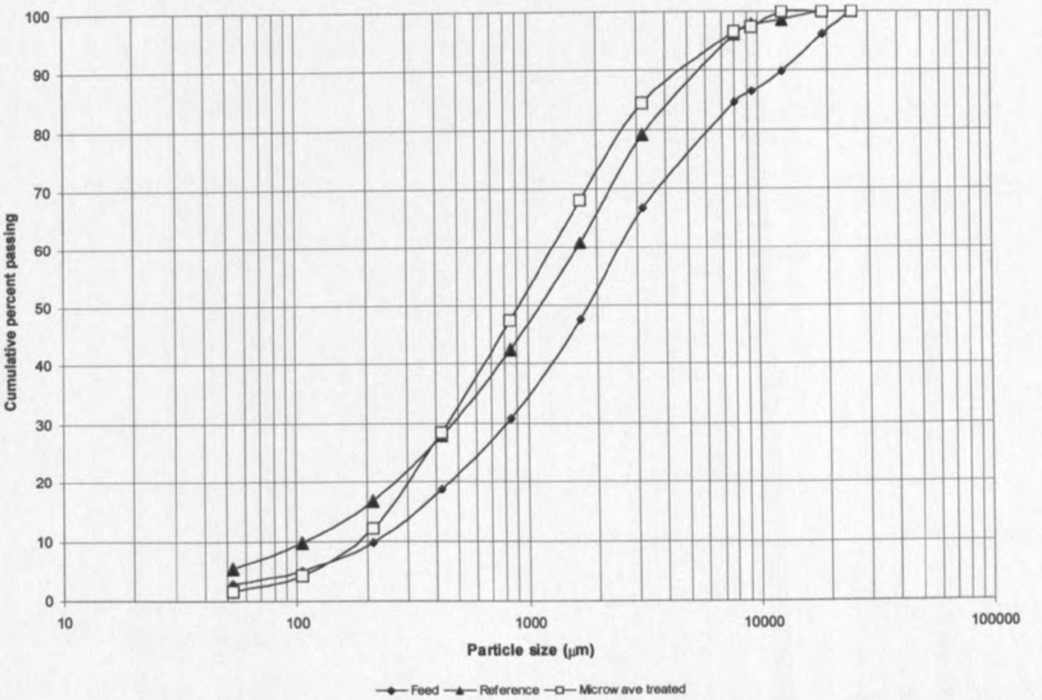


Figure 10.3 Size distribution of as-received and microwave treated P-8 coal from Barmac trials (3000rpm)

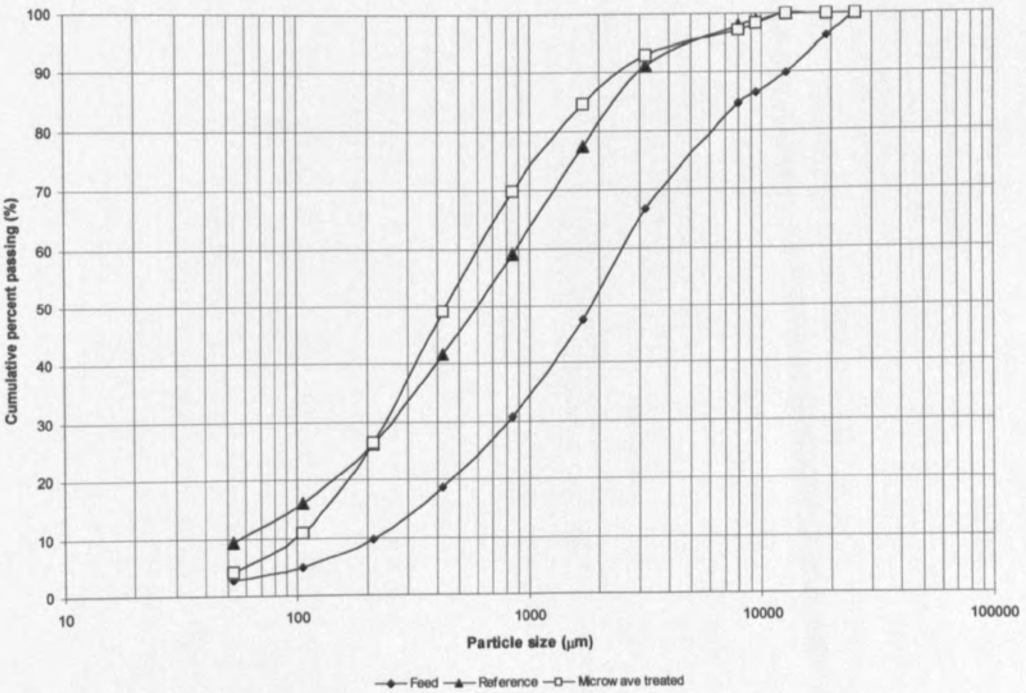


Figure 10.4 Size distribution of as-received and microwave treated P-8 coal from Barmac trials (5000rpm)

The Barmac mill trials demonstrated that microwave treatment can be applied as a method of aiding milling on significant quantities of coal. However, the reduction in grinding resistance of the pilot-scale coal appears to be less than that of laboratory rod mill tests (Figure 10.4). A possible explanation is that the Barmac mill supplies enough energy to a particle (by impacting upon the mill wall or other particles) to split the particle at their weakest points, generating two large particles and a quantity of fines from the impact region. It is speculated that microwave treatment produces or propagates flaws within the structure, increasing the number of weakness points within the structure. Hence, upon impact, particles may produce fewer fines and more similar-sized coarser particles of intermediate size range.

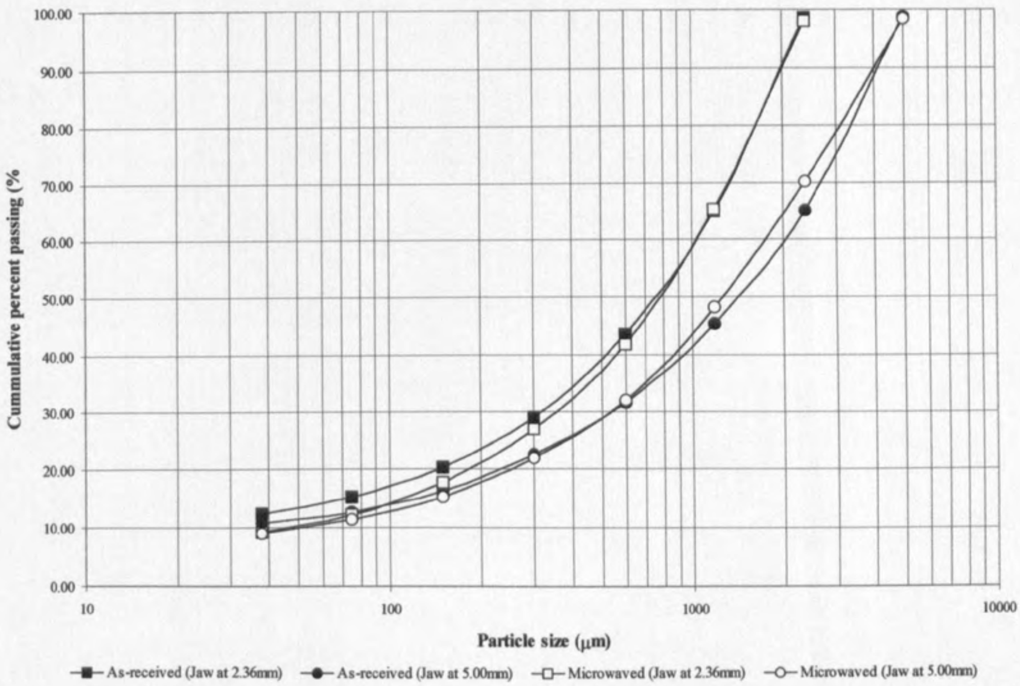


Figure 10.5 Effect of microwave radiation on Jaw Crusher performance (P-8 coal)

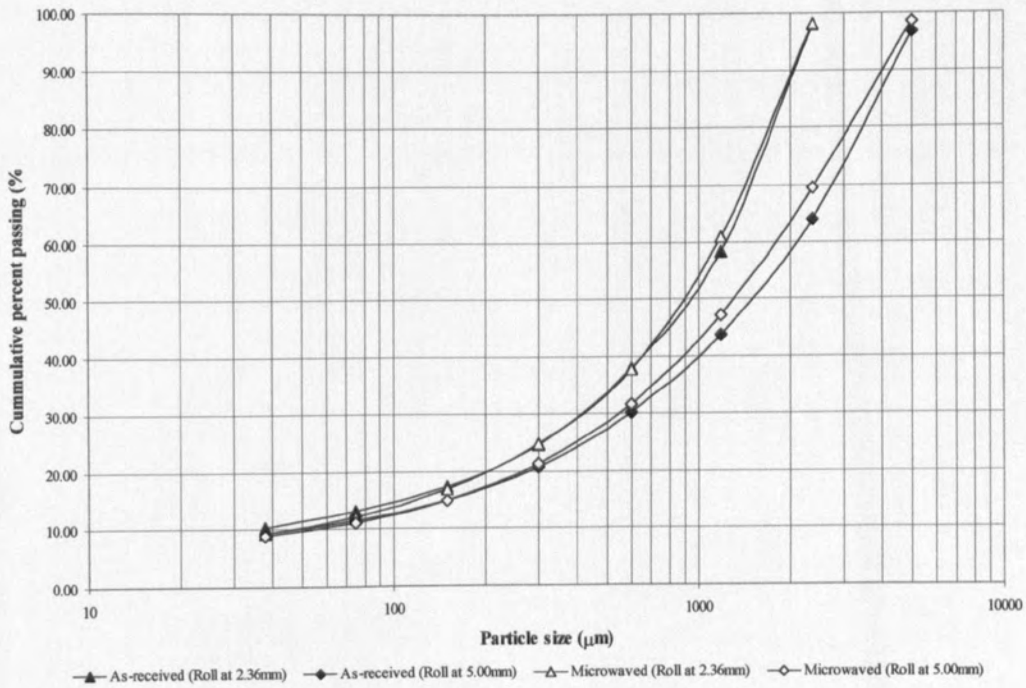


Figure 10.6 Effect of microwave radiation on Roll Crusher performance (P-8 coal)

Clearly the method of size reduction (impact, attrition, compression etc.) significantly affect any comparison between as-received and microwave treated coal. Rod milling is attributed to both impact and attrition milling and results imply that P-8 coal grindability is considerably improved with prior microwave treatment (Figure 10.2). The Barmac crusher relies upon particle on particle impact and pilot-scale results show some selective improvements in P-8 coal grindability. Further tests have been carried out upon microwave treated and as-received P-8 coal by the University of Nottingham using a (Dodge) jaw crusher and roll crusher with product discharge limited to 2.36mm and 5mm respectively. Size distributions were taken for ~1kg representative batch samples following comminution. A finer product was achieved with samples presented to both crushers with discharge setting of 2.36mm irrespective of microwave treatment. Results (Figure 10.5) show that microwave treatment had little effect upon the grindability of P-8 coal within a jaw crusher with similar size distributions to as-received coal. Similarly, results (Figure 10.6) also show little difference between the product size distributions of microwave treated and as-received samples following comminution in a roll crusher. However, results from both jaw and roll crushers show slight reduction in fines production from microwave treated samples. Both machines utilise compression for size reduction. Clearly the type of mill or crusher used is of significant importance to an economic feasibility study and further work would be required upon a more suitable PF generating mill i.e. vertical spindle or lopulco mill.

10.6 Economic considerations

The Barmac mill is not used for PF production in power stations and the use of a PF generating mill i.e. vertical spindle mill, would clearly be a more accurate comparison of the feasibility of microwave treated coal grindability. However, despite the fact that neither the microwave cavity or the mill were operated entirely under optimum conditions, the energy required for microwave treatment was 220kWh/t, far greater (milling energy estimated as 22kWh/t) than that required to produce PF size characteristics from the milling of as-received coal. Furthermore, the microwave treatment energy was based upon power input to load (6.6kW) and not from power draw. The power draw of the pilot-scale cavity corresponding to pilot-scale operating conditions was measured at 12.4kW using a LEM HEME Ltd LH1050 AC/DC Clamp-On Power Meter. The total energy consumption in the microwave stage equates to 414kWh/t. Preliminary work (Chapter 6.11) showed that a reduction in RWI of 50% for the microwave treated sample corresponded to half the batch milling time required to that of a as-received sample (to PF size characteristic). Assuming that RWI can be applied to Bond Work Index (Chapter 4) and using laboratory rod mill results (Table 10.7), 5kWh/t energy would be required at the milling stage. Hence, the total energy requirements for the microwave treated milled coal product is approximately 420kWh/t.

10.7 Pilot-scale conclusions

Pilot-scale testwork was carried out to give a better understanding of scale-up criteria and a more accurate energy balance. The study has demonstrated that microwave processing of coal could be scaled-up. The input power distribution and exposure time for a given load are suggested as being the major limiting factors for the current pilot-scale microwave cavity. Penetration depth is not considered an influencing factor as coal bed depth was maintained less than 0.005m (penetration depth of coals range from 0.2 to 0.6m, Chapter 8.7). The current laboratory and pilot-scale data indicates that the microwave energy required to reduce the resistance to grinding of the coal to any significant degree (i.e. 20% RWI for P-8 coal) far outweighs any energy saving in a subsequent mechanical milling system.

Improvements in cavity design (with regard to energy transfer efficiency and the arrangement of high electric field strength) may reduce the energy requirement considerably. However, it is considered that the use of microwave energy as a means of weakening the coal prior to milling may not prove an economically viable proposition.

CHAPTER ELEVEN

MISCELLANIOUS BENEFITS FOR COAL MICROWAVE TREATMENT

11.1 Introduction-Miscellaneous benefits for coal microwave treatment

Previous workers (Chapter 5) have reported the application of microwave radiation as a potential method of coal desulphurisation and rapid coal pyrolysis. These applications and microwave treated-coal flow results are presented within this study.

11.2 Microwave desulphurisation of coal

The heating rate of pyrite was measured and calculated from dielectric measurements (Chapter 8) at approximately 1°C/s at 0.65kW input power (2.45GHz). Clearly, during preliminary coal microwave heating tests (Chapter 6,7,9 and 10) inherent pyrite would reach temperatures above that required for oxidation (300°C)(Figure 11.1). As shown (Figure 11.1), gaseous sulphur may be released prior to overall coal combustion (all coals tested remain below 250°C), after 8 minutes microwave exposure and providing the heating rate remains constant (1°C/s at 0.65kW input power, Chapter 8), discrete pyrite particles may reach temperature exceeding 480°C. Pyrite changes phase releasing sulphurous gas and reducing the total sulphur content of the coal. Previous work (Zavitanos 1981, Butcher 1995, Bluhm 1980) has demonstrated that this 'altered'pyrite has an increased magnetic susceptibility (in the order of two magnitudes greater) and could be reduced by low gradient magnetic separation.

Figure 11.1 Sulphur release from coal prior to combustion within a microwave field

Initial experiments were carried out to give a better understanding of the nature and occurrence of pyrite in the coal samples and to determine the occurrence of pyrite phase change during microwave heating. Further consideration is presented on the effect of microwave treatment and magnetic separation of the sulphur content of coal.

11.2.1 The nature and mode of occurrence of pyrite in coal

A sample of P-8 coal was examined by Rio Tinto Technology Development to ascertain the occurrence and morphology of pyrite inclusions in coal.

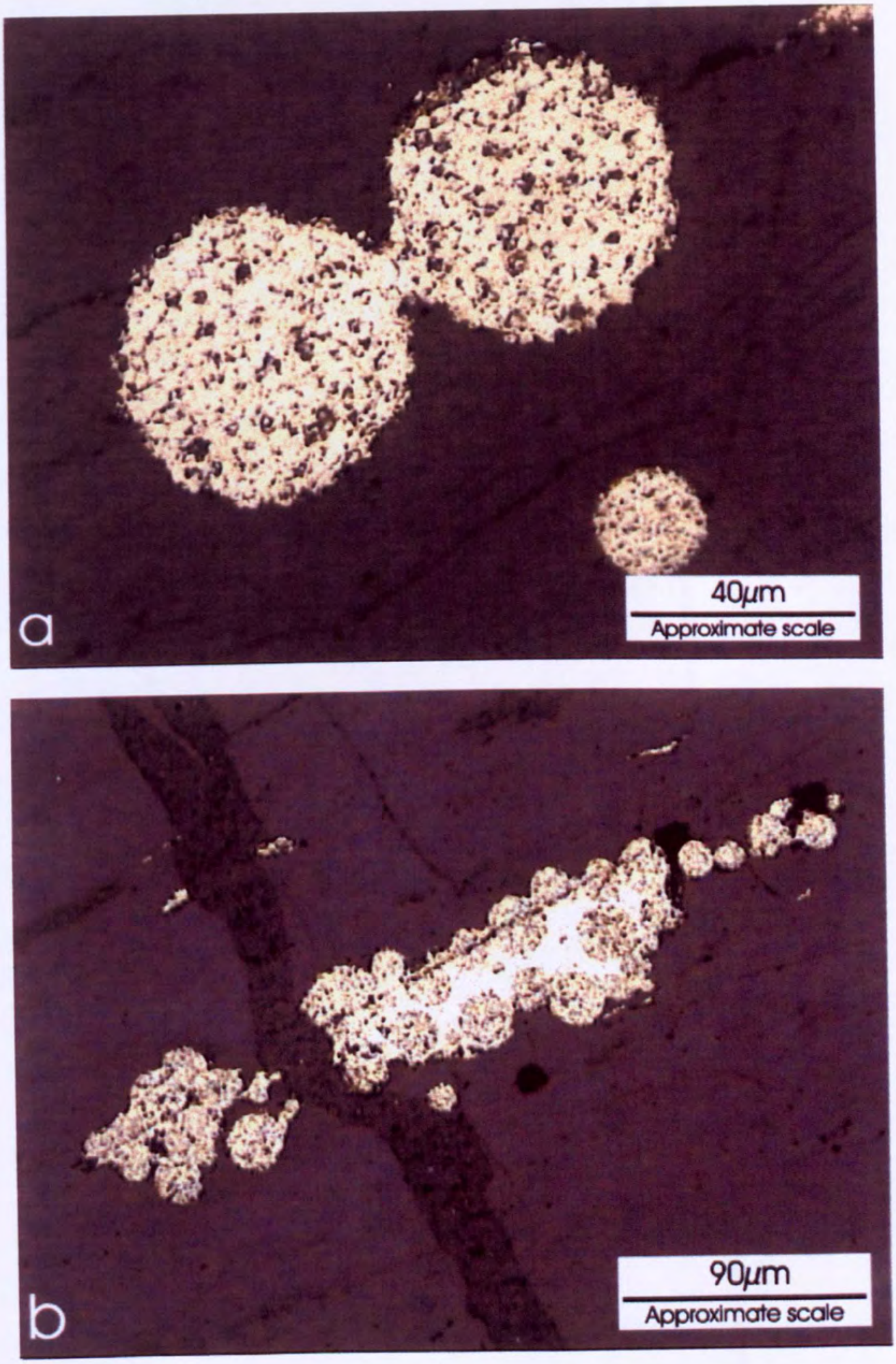


Figure 11.2 Reflected light photomicrograph illustrating pyritic framboids in coal

Relatively few fragments of macroscopic pyrite particles (greater than 1mm) were observed with the aid of binocular microscope. However, microscopic examination shows the presence of a small quantity of pyritic inclusions that rarely exceed 15-20μm. With the use of reflected light microscopy and photomicrographs the microscopic pyrite can be shown to be present in two forms.

One variety of pyrite is present in the form of framboids (spherical bodies) consisting of micrometre-sided bodies of pyrite that are closely packed together to form the larger spherical body (Figure 11.2a). The framboids were found to occur as discrete bodies or small clusters that are widely disseminated throughout the coal. Polyframboid chains (Figure 11.2b) were also present consisting of a number of individual framboids attached together. The area between framboid structures appears to have been filled at a later geological stage by pyrite precipitate. There was a highly variable degree of abundance of this form of pyrite within the P-8 coal tested with the framboids being most abundant along certain bedding planes. These types of pyrite develop by in-situ growth and may have formed at low temperatures by reactions between iron solutions and H_2S gas that could have been generated by decomposing organic debris under reducing conditions.

The other major form of pyrite found within the P-8 coal was associated with the filling of voids within the coal structure with pyrite precipitate (Figure 11.3). The pyrite precipitate structures are of more massive and structureless variation deposited between collapsed wall structures. It can also be seen that the cellular structures in some areas have completely collapsed and contain cell walls fragments only (Figure 11.3b).

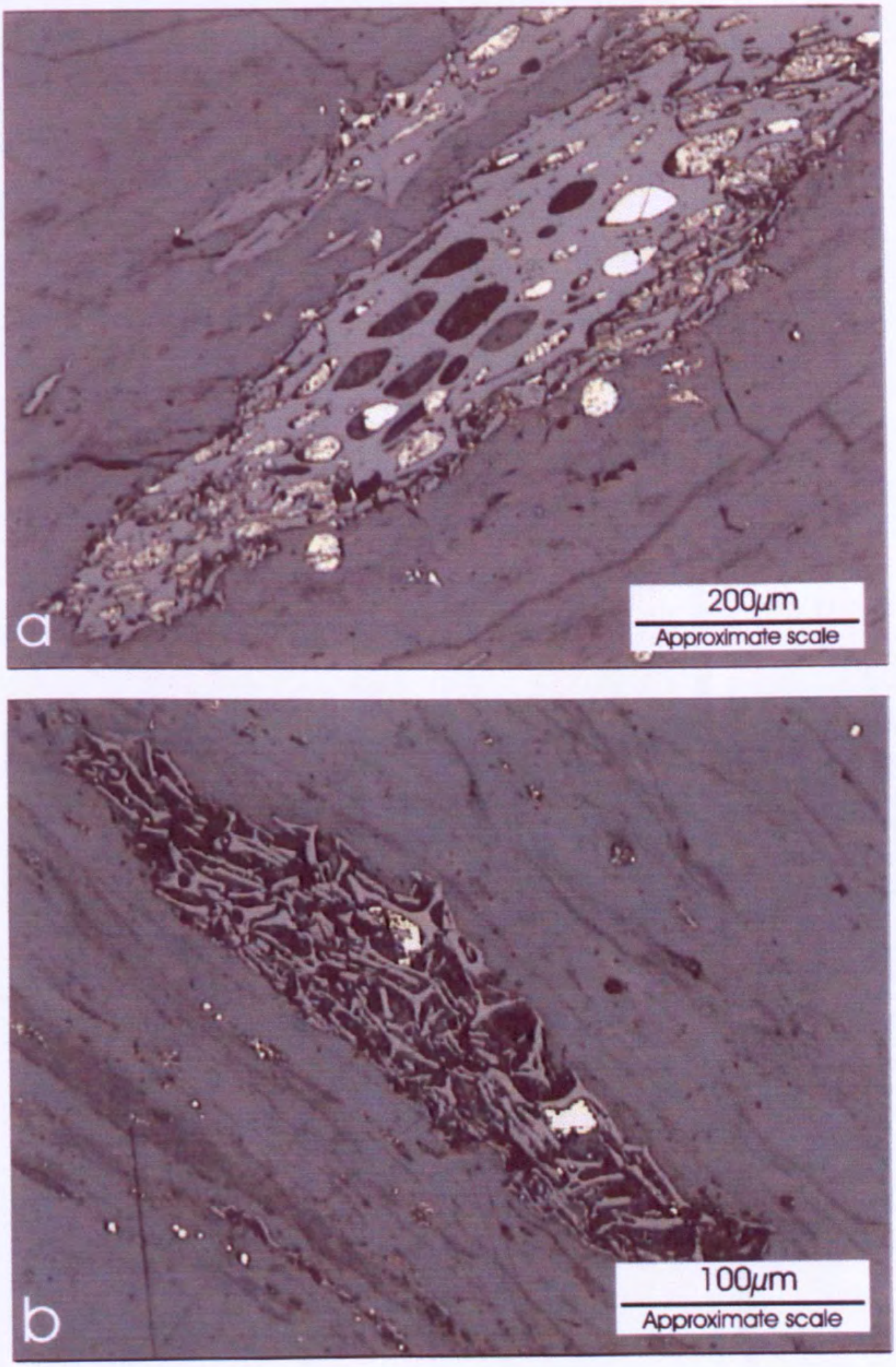


Figure 11.3 Reflected light photomicrograph illustrating coal in which some collapsed cells have been partially filled by pyrite

This form of pyrite also presumably forms under relatively low temperatures and becomes precipitated within open spaces within the cells of the coal. The pyrite is assumed to have formed in a similar manner to that of the framboidal pyrite with variations in depositional conditions accounting for the different morphology (Reynolds 2000).

The overall abundance of pyrite precipitates is highly variable and is completely absent in some coal particles. In other cases this form of pyrite was found to occur near framboidal pyrite and can be relatively abundant locally (Figure 11.4).

The investigation shows that this coal contains a relatively small quantity of pyrite and the pyrite is typically present in extremely fine grains usually in localised areas and bedding planes.

Figure 11.4 False colour, backscattered electron images illustrating the appearance of pyrite that has been precipitated within former openings in coal

11.2.2 Pyrite phase change mechanism

Three 50g (<2mm) 99% pyrite samples were exposed to microwave radiation at 1.5kW for 30s, 120s and 120s (covered in fine particulate feldspar) respectively. The samples were subsequently tested by means of false coloured scanning electron micrograph image analysis to determine the effect of microwave exposure and any oxidation reactions (Reynolds 2000) during heating. The bulk of the pyrite samples appear relatively unaltered indicating that most of the microwave energy is reflected from the pyrite surface. To compensate for this and to simulate inclusion distribution within the coal, a sample was covered with microwave transparent feldspar. In consequence, oxidation appeared to be more extensive than that for the uncovered samples.

Previous studies have concluded that the earlier stages of heating (300°C) result in a decomposition of mineral pyrite (FeS_2) to form pyrrohotite ($Fe_{1-x}S$) as expressed in Equation 11.1. Further heating in the presence of oxygen result in total oxidation as expressed in Equation 11.2.

$$2FeS_2 + 2O_2 \rightarrow 2FeS + 2SO_2 \qquad (11.1)$$

$$4FeS + 7O_2 \rightarrow 2Fe_2O_3 + 4SO_2 \qquad (11.2)$$

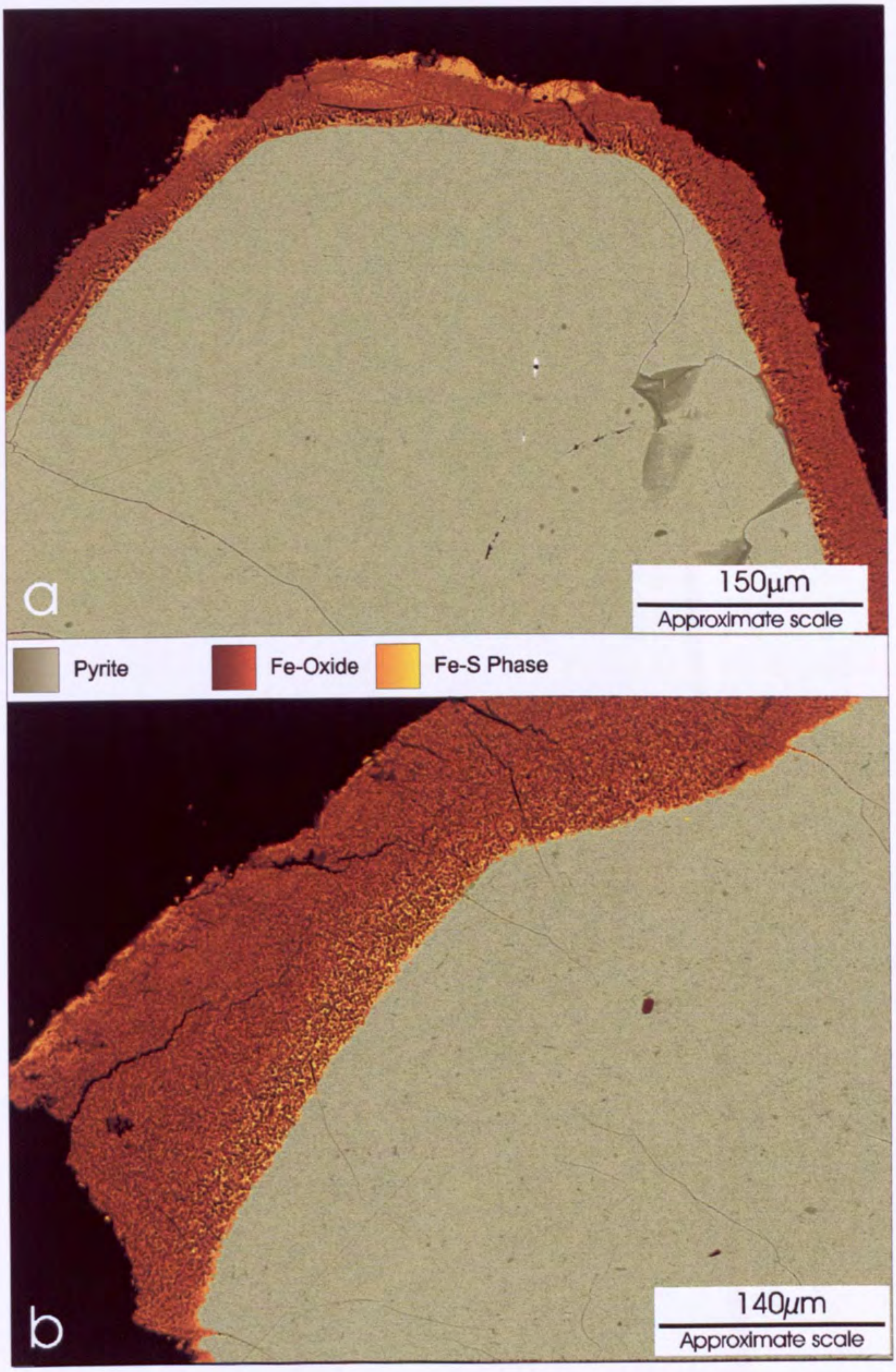


Figure 11.5 Pyritic oxidation (Pyrite under atmospheric conditions, 30s microwave exposure at 1.5kW, 2.45GHz mono-mode cavity)

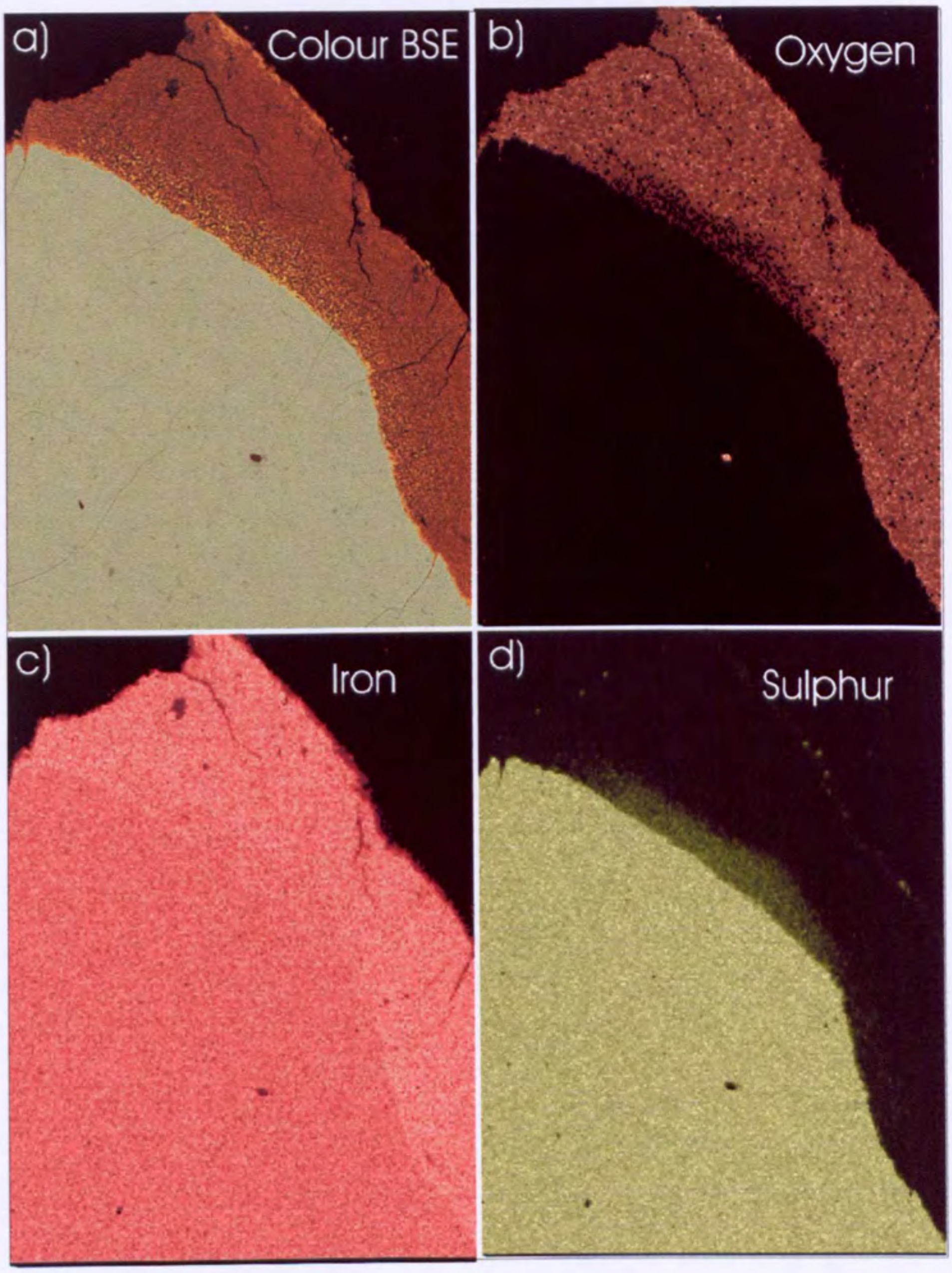


Figure 11.6 Pyritic oxidation, x-ray dot imaging (Pyrite under atmospheric conditions, 30s microwave exposure at 1.5kW, 2.45GHz mono-mode cavity)

The uncovered microwave exposed samples shows iron-oxide predominantly as opposed to an FeS phase suggesting rapid oxidation (Figures 11.5 and 11.6). It would also appear that the Fe-oxide diffuses into the pyrite particle whilst sulphur diffuses toward the surface, as illustrated in Figure 11.7.

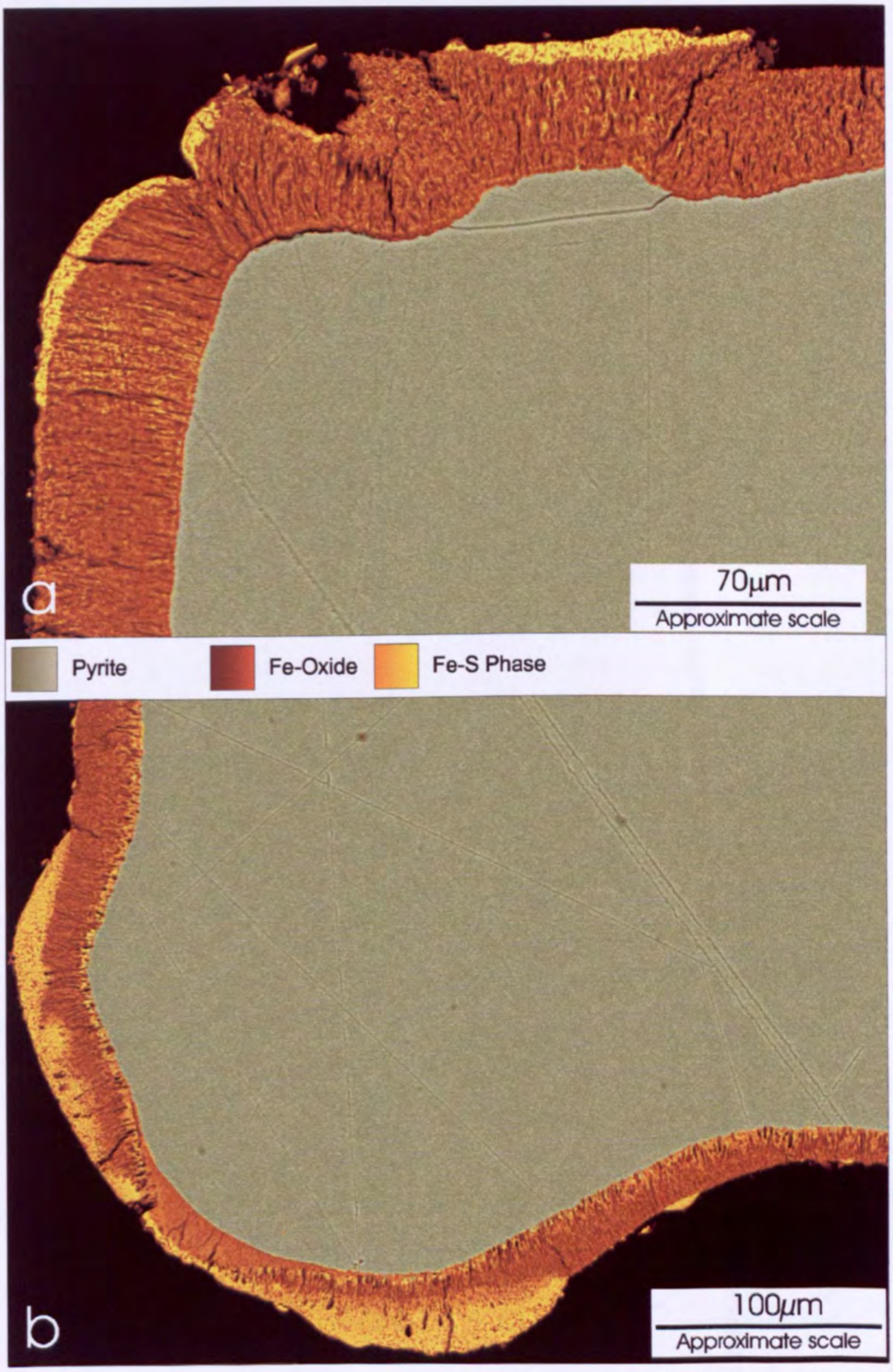


Figure 11.7 Pyritic oxidation (Pyrite under atmospheric conditions, 120s microwave exposure at 1.5kW, 2.45GHz mono-mode cavity)

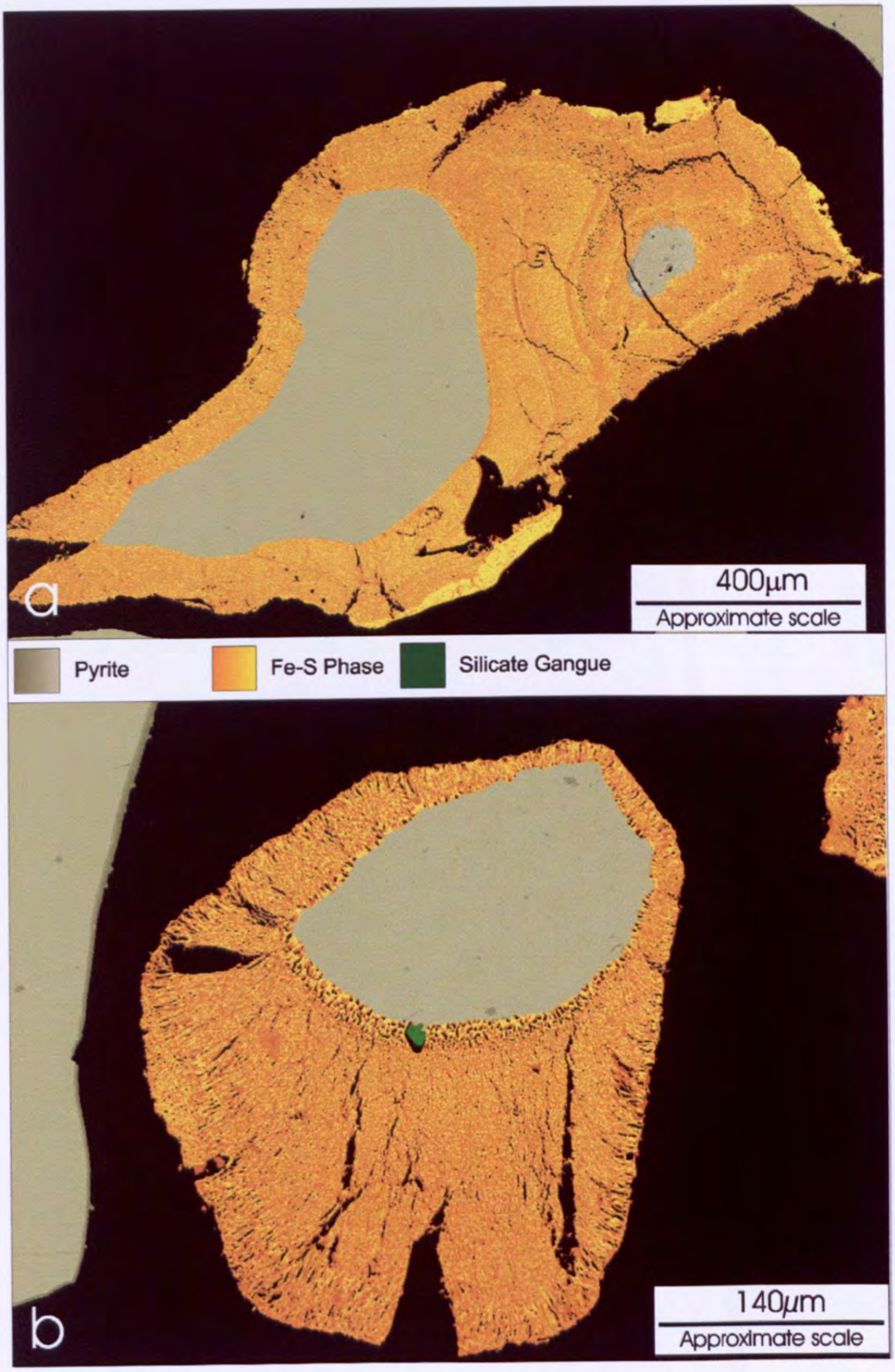


Figure 11.8 Pyritic oxidation (Pyrite covered in sand under atmospheric conditions, 120s microwave exposure at 1.5kW, 2.45GHz mono-mode cavity)

The degree of alteration appears marginally more extensive with the feldspar covered sample. Furthermore; Fe-oxide-the dominant product in previous samples-is absent and the particle surface consists primarily of FeS (Figure 11.8). The fine-grained feldspar has had some effect on the ability of the sample to oxidise, possible restricting the availability of oxygen and may lower the heating rate. This situation is probable with regard to pyrite inclusions within coal and suggests the possibility of preferential desulphurisation prior to combustion with microwave radiation.

11.2.3 Sulphur reduction by magnetic separation of microwave treated coal

Three coals (prepared using cone, quartering and riffling methods) were used in a microwave desulphurisation feasibility study; coal sulphur contents are given in Table 11.1. Samples were irradiated (0.65kW, 2.45GHz) for 5 minutes and provided that the heating rate of pyrite remained relatively constant with increasing temperature, the final temperature of the pyrite would be in the range between 300°C to 350°C.

Table 11.1 Forms of sulphur, feed samples (dry basis)

Sample	Pyritic sulphur (%)	Sulphate sulphur (%)	Organic sulphur (%)	Total sulphur (%)
P-3	0.67	0.12	0.82	1.71
P-6	2.04	0.40	0.97	3.41
P-2	0.52	0.05	0.90	1.47

The coarse sub-samples (500g) were tested using a 1.2Tesla magnetic field strength, Boxmag Rapid dry high intensity Magna roll separator. Fine coal samples (100g) of <200 μm particles were separated using a Boxmag Rapid wet high intensity magnetic separator (1.6 Tesla magnetic field strength).

Forms of sulphur analyses (coarse samples only) show that the greatest quantities of pyritic sulphur content were detected in the magnetic product from the tests irrespective of microwave exposure (Tables 11.2 and 11.3). Analysis of microwave treated magnetic product shows substantially less pyritic sulphur than the as-received samples suggesting some gaseous sulphur loss has occurred.

Table 11.2 Forms of sulphur, as-received samples after dry Magna Roll magnetic separation (dry basis)

Sample	Pyritic sulphur (%)	Sulphate sulphur (%)	Organic sulphur (%)	Total sulphur (%)
P-3, non-magnetic	0.26	0.08	1.31	1.65
P-3, magnetic	2.27	0.1	0.09	2.46
P-6, non-magnetic	1.86	0.33	0.63	2.82
P-6, magnetic	2.94	0.31	0.78	4.03
P-2, non-magnetic	0.14	0.05	1.18	1.37
P-2, magnetic	1.95	0.09	0.55	2.59

Table 11.3 Forms of sulphur, microwave treated (0.65kW, 2.45GHz) samples after dry Magna Roll magnetic separation (dry basis)

Sample	Pyritic sulphur (%)	Sulphate sulphur (%)	Organic sulphur (%)	Total sulphur (%)
P-3, non-magnetic	0.25	0.06	1.13	1.44
P-3, magnetic	0.2	0.05	1.44	1.69
P-6, non-magnetic	0.58	0.1	0.8	1.48
P-6, magnetic	1.08	0.12	0.71	1.91
P-2, non-magnetic	0.14	0.04	1.01	1.19
P-2, magnetic	1.28	0.12	0.56	1.96

The results for coarse coal samples separated using a Dry Magna Roll separator (Boxmag Rapid) are summarized in Table 11.4 (as-received samples) and Table 11.5 (microwave treated samples).

Table 11.4 Magnetic separation (Dry Magna Roll Separator) as-received coals (Boxmag Rapid)

Sample	Mass retained (g)	Mass retained (%)	Ash content (%)
P-3, original	583.8	-	-
P-3, non-magnetic	450.5	87.5	13.1
P-3, magnetic	64.1	12.5	17.3
P-6, original	514.6	-	-
P-6, non-magnetic	415.7	71.2	8.3
P-6, magnetic	168.1	28.8	28.4
P-2, original	499.5	-	-
P-2, non-magnetic	452.0	90.5	14.0
P-2, magnetic	47.5	9.5	35.9

Table 11.5 Magnetic separation (Dry Magna Roll separator) microwave treated (0.65kW, 2.45GHz, 108kWh/t) (Boxmag Rapid)

Sample	Mass retained (g)	Mass retained (%)	Ash content (%)
P-3, original	486.7	-	-
P-3, non-magnetic	385.6	81.6	17.4
P-3, magnetic	87.0	18.4	17.2
P-6, original	472.6	-	-
P-6, non-magnetic	369.4	75.9	16.2
P-6, magnetic	117.3	24.1	25.1
P-2, original	490.4	-	-
P-2, non-magnetic	445.2	90.8	14.2
P-2, magnetic	45.2	9.2	33.6

The mass retained for as-received and microwave treated coals for both magnetic and non-magnetic concentrates are given in Tables 11.4 and 11.5. There was little change in mass percentage recovery following microwave treatment and due to the particle size and the nature of pyrite phase change it is difficult to access the extent of microwave treatment on desulphurisation. The mineral ash components (ankerite, apatite and siderite) are paramagnetic and may also be present in the magnetic concentrate. This may account for the increase in ash mineral content of the magnetic concentrate. However, results (Tables 11.2 too 11.5) show that magnetic separation reduces the pyritic content of coal irrespective of microwave treatment.

Subsequent tests were carried out with samples of fine size characteristics (-212mm). For coals and products separated using a (1.6 Tesla magnetic field strength) Boxmag Rapid wet high intensity separation technique, all the products were subsequently filtered and dried. The total sulphur content of the samples was measured using a 'LECO SC-132 Sulphur Determination Unit'.

Table 11.6 High Intensity Wet Magnetic separation (Expanded Metal Matrix) as-received coals (Boxmag Rapid)

Sample	Mass retained (g)	Mass retained (%)	Ash content (%)	Total sulphur (%)
P-3, original	88.6	-	-	1.94
P-3, non-magnetic	82.0	90.3	10.8	1.79
P-3, magnetic	8.6	9.7	39.6	2.65
P-6, original	97.6	-	-	3.88
P-6, non-magnetic	76.7	78.6	13.8	3.12
P-6, magnetic	20.9	21.4	20.2	4.66
P-2, original	99.5	-	-	1.62
P-2, non-magnetic	92.2	92.7	7.7	2.79
P-2, magnetic	7.3	7.3	25.2	1.43

Results (Table 11.6) for the as-received coals following magnetic separation indicate that some pyrite can be removed by wet separation. Furthermore, there was some evidence of increased mineral matter content in the magnetic concentrates suggesting a possible method of de-mineralisation.

Table 11.7 High Intensity Wet Magnetic separation (Expanded Metal Matrix) microwave treated (0.65kW, 2.45GHz, 108kWh/t) (Boxmag Rapid)

Sample	Mass retained (g)	Mass retained (%)	Ash content (%)	Total sulphur (%)
P-3, original	68.7	-	-	1.86
P-3, non-magnetic	62.1	90.4	13.0	1.56
P-3, magnetic	6.6	9.6	53.4	1.88
P-6, original	91.0	-	-	1.81
P-6, non-magnetic	68.4	75.2	6.6	1.55
P-6, magnetic	22.6	24.8	13.5	2.06
P-2, original	107.6	-	-	1.45
P-2, non-magnetic	86.5	80.4	8.71	1.27
P-2, magnetic	21.1	19.6	25.64	2.00

Following microwave exposure (0.65kW, 2.45GHz for 5 minutes) and subsequent magnetic separation, results (Table 11.7) confirmed increased mineral matter contents in magnetic concentrates in comparison to those values for as-received coals. This may be due to paramagnetic and ferromagnetic inclusions. The total sulphur content decreases in non-magnetic concentrates as a result of microwave treatment for the coals tested and there is evidence that some sulphur is removed as a gas prior to magnetic separation (Table 11.6 and 11.7). Clearly, not all pyrite would change phase to FeS, some pyrite may oxidise further (iron oxides) and some may not oxidse and report to the magnetic concentrate. Furthermore, initial tests show that not all the pyrite changes phase, (Figure 11.8) particles may report to the magnetic concentrate with enhanced magnetic properties from partial surface (FeS) phase change. Results indicate that each coal may respond differently to magnetic separation and, in some cases, microwave treatment may improve pyrite separation. Further work is required to fully access the limitations and feasibility of this method of desulphurisation.

11.3 Rapid coal pyrolysis

Figure 11.9 Coal pyrolysis after microwave heating

During this study no attempt was made to quantify or investigate microwave coal pyrolysis applications. However, in the microwave heating of some coals there is evidence of coal pyrolysis (Figure 11.9). This is usually associated with pyrite inclusions within the proximity of the affected coal and is possibly the result of locally high temperatures during heating. During the current study, bulk coal temperatures did not reach those required for coal pyrolysis (500-700°C) and the technique may prove to be uneconomical due to the poor dielectric heating rate of coal (Chapter 8). However, further work is required to investigate whether doping the coal (within an inert atmosphere) with a material of high dielectric heating rate may initiate coal pyrolysis at lower energy requirements.

11.4 Microwave treated coal flowability

The effect of microwave treatment on coal flowability was studied on P-1 and P-6 coals. Representative samples of both coals were exposed to microwave radiation (173kWh/t) and allowed to cool to ambient temperature before containment. As-received and microwave-treated coal flowability was evaluated using the Johanson Indicizer test at CRE Group. In particular, the 20^0 Arching Index for as-received and microwave treated samples were determined at increasing coal moisture contents (Figure 11.10).

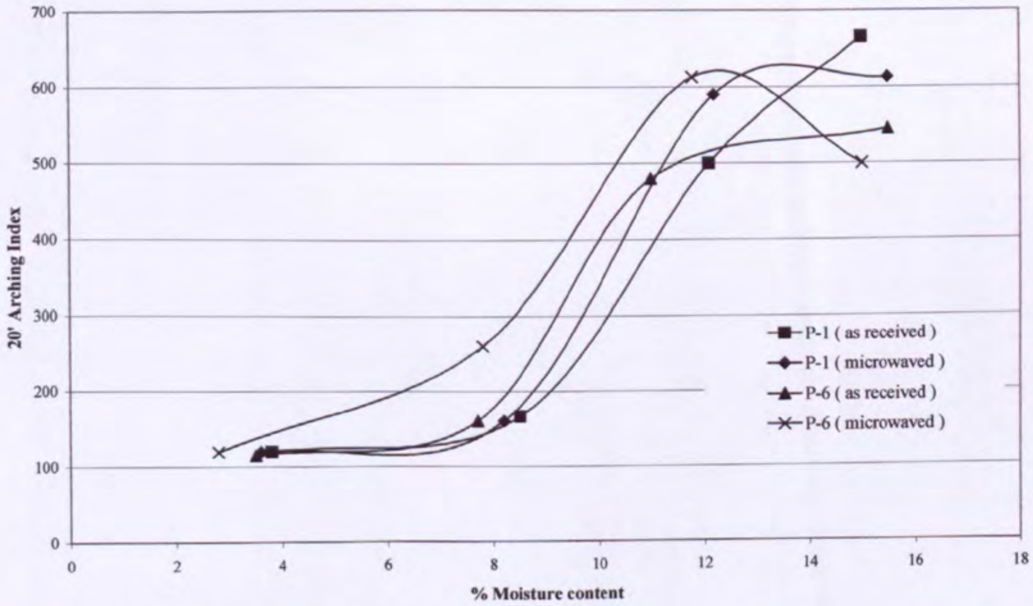


Figure 11.10 Johanson Indicizer tests for as-received and microwave treated P-1 and P-6 coals

Analysis suggests that microwave pretreatment may alter the flowability of coal. The maximum 20^0 Arcing indices for microwave-treated coal samples were achieved at moisture levels of 12% and 13.5% for P-6 and P-1 coals respectively. Maximum 20^0 Arching indices for the as-received samples were not evident at moisture contents below 15%. The Johanson test shows similar results for microwave and as-received P-1 coal initially with as-received samples exhibiting a higher 20^0 Arching Index at increased moisture levels due to the microwaved sample reaching a maximum. In contrast, microwave treated P-6 coal exhibited an increasing 20^0 Arching Index at lower moisture levels compared to as-received P-6 coal. Results (Figure 11.10) for P-6 coal also shows that P-6 microwaved coal exhibited a lower 20^0 Arching Index at moisture levels greater than 14%. A possible explanation may be associated to particle surface area which increases following microwave exposure (Chapter 7). The generation of surface cracks on coal by microwave radiation may affect particle flowability as the particles may be capable of retaining greater quantities of moisture; forming 'bridges' to other particles. Hence, the microwave treated coal would become 'sticky' at lower moisture levels than as-received coal. The effect of microwave treatment on coal flowability would therefore depend on the generation of cracks produced by the heat treatment. It would be expected that the greater the quantity of surface cracks produced by pretreatment the more significant the deviation in flowability characteristics from the as-received coal.

11.5 Conclusion- Microwave beneficiation to improve handleability

Experimental work has shown that the use of microwave treatment may have some use in partial desulphurisation of coals with high pyrite contents. In particular, the potential mechanism for partial oxidation has been discussed and shows that not all the pyrite inclusion need be oxidised for magnetic separation. Mineralogical analysis has indicated that not all coal particles contain pyritic inclusions and shows the average grain size of pyrite inclusion were between 20-40μm for the tested coal. Further work would be needed with selecting suitable coals, which may benefit from microwave pyritic reduction and the optimisation of a microwave enhanced magnetic separation process. In particular the feed size, pyrite particle size, microwave operating conditions and magnetic separation conditions require further investigation to gain a better understanding of this method of desulphurisation.

Fundamental studies have shown that there is a significant change in the flow characteristics of coals following microwave exposure. The possibility may exist for improved coal flow and further work is necessary to access the viability of such a process.

Coal pyrolysis has been observed during microwave tests, previous work has suggested the use of microwave energy as a method of rapid coal pyrolysis (Monsef-Mirzai 1995) and may present a method of coal removal from shale tailings.

CHAPTER TWELVE

CONCLUSION AND RECOMMENDATIONS FOR FURTHER WORK

12.1 Conclusions and recommendations for further work

Experimental results (Chapter 6 and 7) have shown that microwave irradiation can significantly affect the grindability of coals. Results have shown a 40-50% reduction in Relative Work Index, particularly for low rank coals (Chapter 7). Fundamental studies indicate that each individual coal has an optimum microwave energy requirement for maximum improvement in coal grindability. Analysis (Chapter 6 and 7) suggest that coal composition, in particular, mineral matter and inherent moisture contribute to the mechanisms responsible for producing the weaknesses within the coal structure during microwave exposure. The differential heating rates and associated expansion rate of the constituent mineral matter in coal is believed to be the principle mechanism for microwave-improved grindability (Chapter 9). Results (Chapter 6) have also shown that microwave grindability also depends upon initial particle size and feed size distribution; but factors may effect mill performance. Microwave input power, sample load and exposure time may be varied with little change in the reduction in RWI at constant energy per unit mass (Chapter 10).

Dielectric measurement (Chapter 8) has shown that electric permittivity decreases with coal rank (dry, mineral free basis). However, the moisture and mineral matter content may increase the bulk electric permittivity of the coal. Permittivity is relatively high for low rank coals as a result of the increased inherent moisture levels for such coals. The relative real permittivity of coal is greater than that of many of the mineral matter forms associated with coals. However, one exception is pyrite which if present in sufficient quantity, may increase the bulk permittivity of coal. Dielectric properties and mineralogical composition could be used to indicate how a coal would respond to microwave treatment i.e. in terms of heating rate and changes in grindability.

Pilot-scale tests demonstrated that the microwave treatment process could be scaled up providing a better understanding of the process. Results (Chapter 10) showed some improvement between the 5 to 0.5mm size range and generated less fines during Barmac Rock on Rock tests. Mill performance varied depending upon the predominant breakage mode in the different mills and the extent of microwave treatment of the coals. Any further tests should take into consideration the mill design and operating features. An economic evaluation based upon the pilot-scale data and rod mill grindability tests indicated that a gross energy input of 420kWh/t was required for the microwave treatment. This current economic balance is not favourable in comparison to mill energy requirements of 20-40kWh/t for PF generating mills in industry (Spiers 1950). However, the microwave treatment process has not been optimised and further work is necessary in this field.

The current tests were carried out using multi-mode microwave cavity units which exhibit low electrical to microwave power conversion efficiencies. In particular, the pilot-scale microwave unit required 12.4kW power draw to supply 6.6kW to the cavity. Higher efficiencies (80-90%) may be possible using mono-mode cavity designs and tests are needed to determine if grindability improvements can be duplicated or enhanced within such cavities. The factors which influence the microwave power absorption include, (i) the choice of a coal with a high loss factor and (ii) increased electric field strength distribution possibly at lower applied frequencies. For current tests, the applied frequency was 2.45GHz, dielectric measurements (Chapter 8) show little variation of the dielectric properties of coal with increased frequency. However, penetration depth decreases with increasing frequency and appropriate sample presentation may become necessary. Cavity design has a significant influence on power absorption and a specification meeting the requirements of coal treatment may result in an improvement in the efficiency of coal microwave processing and reduce capital and operating costs.

The current study has also demonstrated the potential use of microwave energy as a method of coal desulphurisation. Further work is required to investigate the feasibility and optimisation of this technique for the desulphurisation and demineralisation of coal.

NOMENCLATURE

A	Coefficient of absorption (F/m^2)
α_v	Coefficient of volumetric expansion (K^{-1})
c	Speed of light (m/s) 3×10^8 m/s
C_p	Specific heat capacity (J/kg)
d	Diameter of the rod (m)
D	Distance (m)
$\mathrm{D_p}$	Penetration depth of wave (m)
$\mathrm{D_s}$	Skin depth (m)
D	Diameter of the mill (m)
D_1	Initial particle diameter (m)
D_2	Final particle diameter (m)
δf	Change in centre frequency when sample is inserted (Hz)
e	Napierian logarithm base (dimensionless) 2.7182818285 (10dp)
E	Electric field strength (v/m)
$\mathrm{E_{insitu}}$	Electric field strength within the material (average) (v/m)
$\mathrm{E_{ext}}$	Electric field strength of microwave cavity (v/m)
E_{nergy}	Energy input (kWh/t)
ε_0	Permittivity of free space (F/m)
ε^*	Complex permittivity (F/m)
ε'	Real permittivity (F/m)
ε''	Imaginary permittivity (F/m)
ε_r'	Relative dielectric constant or relative real permittivity (dimensionless)
ε_r''	Relative loss factor or relative imaginary permittivity (dimensionless)
f	Frequency (Hz)
f_0	Resonant centre frequency (air filled) (Hz)
F	80% passing sizes of the feed (μm)
Fn	80% passing size of the feed coal, non-treated coal (μm)
Ft	80% passing size of the feed coal, microwave treated coal (μm)
G	Young's modulus (N/m^2)
γ	Surface energy per unit surface area (J/m^2)
H	Magnetic field strength (N/A^2)
HGI	Hardgrove Grindability Index (dimensionless)
j	$\sqrt{-1}$ (imaginary complex number) (dimensionless)
$J_1(function)$	First order Bessel function (Perry 1984) (dimensionless)
l	length of crack (m)
λ	Wavelength (m) $\lambda=c/f$
m_x	Mass fraction of component x
M	Mass of the body heating (kg)
Mn	80% passing size of the milled coal, non-treated coal (μm)
Mt	80% passing size of the milled coal, microwave treated coal (μm)

μ_0	Permeability of a vacuum (N/A^2)
μ''	Permeability (N/A^2)
N	Number of moles in fluid (kmol)
O_c	Dielectric conductivity (S/Ω)
P	80% passing sizes of the product (μm)
P	Power absorbed (W/m^3)
P_a	Power absorbed (W)
P_g	Pressure of gas (N/m^2)
Q	Energy absorbed or produced (W)
θ_1	Original temperature (°C)
θ_2	Final temperature (°C)
θ	Change in temperature (^{0}C)
R	Universal gas constant (8.314kJ/kmol K)
RWI	Relative Work Index (%)
r	Radius of particle (m)
r_{cavity}	Radius of the resonant cavity (m)
r_e	Coefficient of reflection (dimensionless)
Rr	Reduction ration (F/P)
ρ	Density of material (kg/m^3)
ρ_x	Density of component x (kg/m^3)
t	Change in time (s)
T_g	Temperature of gas (K)
τ'	Constant approximated as 0.25 (dimensionless)
V	Voltage (v)
V_g	Volume of gas (m^3)
V_1	Original volume (m^3)
V_2	Final volume (m^3)
V	Volume of material (m^3)
V_c	Critical speed (rev/min)
V_{cavity}	Volume of cavity (m^2)
V_{sample}	Volume of sample (m^2)
W	Work input per tonne (kWh/t)
Wi	Bond Work Index (kWh/t)
ω	Angular frequency (s^{-1})
$(x_{l,m})$	Function of the air filled cavity (dimensionless)

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APPENDIX A

SUMMARY OF ANALYSIS TECHNIQUES

Proximate analysis (BS 1016 part 3)

Proximate analysis evaluates a coal in terms of moisture, ash minerals, volatile matter and fixed carbon content of the coal. The coal sample used for proximate analysis is prepared in accord with BS 1017 (particle size less than 212μm).

There are three methods described in BS1016 for determination of the moisture content of a coal: Direct gravitational method, drying in nitrogen and drying in a vacuum, the drying in a vacuum method is described here as the method used within this study.

A sample (1g±0.01g measured to 4 dpi) is heated between 105°C and 110°C within a vacuum. The temperature is maintained for over 40 minutes before the mass of the sample and container are evaluated (note the sample weight is measured while the temperature is maintained at 105°C). The moisture content is derived from the mass balance given in Equation A.1:

$$M_{ad} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 \qquad \text{(A.1)}$$

M_{ad} Percentage of moisture in the analysis sample.
M_1 Weight of the sample vessel.
M_2 Weight of the sample plus the sample vessel before heating.
M_3 Weight of the sample plus the sample vessel after heating.

The volatile matter is defined as the percentage of gaseous product; not inclusive of moisture, during heating under air free-controlled conditions. The volatile matter component takes into consideration the weakly bonded molecules (containing carbon, hydrogen and oxygen) that readily devolatilise during heating.

A pre-weighed sample (~1g±0.01g) is enclosed within a crucible and heated in a muffle furnace at a temperature of 900°C±5°C for exactly 7 minutes. After the heating period the sample is allowed to cool for no longer than 5 minutes at which time it is to be introduced into a desiccator until cool enough to reweigh. The percentage of volatile matter is calculated using the mass balance shown (Equation A.2):

$$M_{vol} = \frac{m_2 - m_3}{m_2 - m_1} \times 100 - M_{ad} \tag{A.2}$$

M_{vol} Percentage of volatile matter in the analysis sample.

The ash component is derived by introducing the a sample (~1g±0.01g) into a muffle furnace at a temperature of 815°C±10°C for 90-120 minutes. The sample is removed and allowed to cool for no more than 5 minutes before being moved to a desiccator until cool enough to weigh. The ash content is determined using Equation A.3:

$$M_{Ash} = \left(1 - \frac{m_2 - m_3}{m_2 - m_1}\right) \times 100 \tag{A.3}$$

M_{Ash} Percentage of gangue ash minerals in the analysis sample.

The fixed carbon content component represents the relative percentage of the strongly bonded carbon based molecules within the coal. The fixed carbon content is calculated as the remaining part of the coal minus the moisture, ash mineral and volatile matter components:

$$M_{FC} = 100 - (M_{ad} + M_{vol} + M_{Ash}) \tag{A.4}$$

M_{FC} Percentage of fixed carbon content in the analysis sample.

Calorific value (BS 1016 part 5)

The calorific value is defined as the number of heat units measured when a unit quantity of fuel is burned in an oxygen rich environment within a bomb under standard conditions. The preferred method of experimental measurement uses an adiabatic bomb calorimeter due to it's simplicity and accuracy, the reader is advised to consult BS1016 part 5 for further details on other methods of determination. The gross calorific value is calculated from relationships between the temperature rise and the mean effective heat capacity of the system immediately before and after total combustion. The crucible, cotton connecting wire and coal sample (~1g±0.01g) are weighed (4dpi) and introduced into the bomb (cotton wire is connected to the firing wire of measured length). The bomb is assembled and purged with oxygen to a pressure of 30bar, the initial temperature of the water within the bomb is measured allowing sufficient time for the stirrer and pump to stabilise the adiabatic conditions within the calorimeter. It must be noted that the initial temperature should be approximately 25°C and recorded to 0.001K. The bomb is fired and final temperature recorded as the maximum increase or after a pre-determined period of time has elapsed. The calorific value is calculated using Equation A.5:

$$Q_{gr,v} = \frac{(\Delta\vartheta)C_{sp5} - e_1 - e_2 - e_3 - e_4}{m} \tag{A.5}$$

$Q_{gr,v}$	Gross calorific value at constant volume (kJ/kg)
C_{sp5}	Mean effective heat capacity of the calorimeter (J/K)
$\Delta\vartheta$	Change in temperature (K)
m	Mass of coal (g)
e_1	Correction for the heat of combustion of the cotton (J)
e_2	Correction for the heat of combustion of the firing wire (J)
e_3	Correction for the heat of formation of sulphuric acid (J)
e_4	Correction for the heat of formation of nitric acid (J)

Ultimate analysis (BS 1016 part 6)

The ultimate analysis or some times referred to as elemental analysis is an analysis of the coal in terms of the carbon, hydrogen, nitrogen and sulphur content (usually carbon dioxide content is reported as well as the elemental constituents). The analysis technique takes into consideration both the organic coal material as well as the inorganic material deposited with the coal. The nitrogen is assumed to be part of the organic coal substance, the total sulphur includes both organic sulphur as well as inorganic sulphates and minerals. The carbon content includes the presence of mineral carbonates as well as in the coal, the hydrogen content includes the hydrogen present in hydrocarbon chains and rings as well as within the moisture and mineral components.

A brief description of the experimental process is described here and the reader is advised to consult BS1016 part 6 for further details.

The carbon and hydrogen contents are measured simultaneously. The coal sample (0.2-0.3g) is combusted at 800°C in an oxygen atmosphere for over 2 hours and the gas is passed over copper oxide. It is assumed that this converts all of the carbon to carbon dioxide and hydrogen to water vapour. The moisture content is assessed simultaneously in accordance to BS1016 part 3. Oxides of sulphur and chlorine are retained in the combustion tube by lead chromate and silver gauze. The water vapour is adsorbed by manganese dioxide, the carbon dioxide by soda asbestos, their relative content is evaluated by a mass balance over the procedure.

The nitrogen content is determined by heating 0.1g of sample with sulphuric acid in the presence of a catalyst to destroy the organic material and convert the nitrogen to ammonium sulphate. The ammonia is removed by the addition of sodium hydroxide and steam distillation with boric acid. The ammonia solution is directly titrated with 0.01N sulphuric acid.

The total sulphur is determined by a high temperature method. The coal sample (0.5g) is combusted at 1350°C in a high current of oxygen, this converts all the sulphur to its oxides which is adsorbed in hydrogen peroxide to form sulphuric acid. The total acidity is determined by titration and content evaluated.

The carbon dioxide content is determined by a process of desolving a sample of coal in hydrochloric acid and adsorbing the carbon dioxide in benzylamine solution. The salt formed is titrated with potassium methoxide in toluene using thymol blue as an indicator.

Forms of sulphur analysis (BS 1016 part 11)

The total sulphur content of a coal is determined as described by BS1016 part 6. The reader is advised that the following is a brief summary of the experimental procedures and should consult BS1016 part 11 for further details.

The sulphate sulphur is determined by desolving ~5g±0.01g of coal (prepared in accordance to BS1017) in 60ml hydrochloric acid just below boiling. The filtered extract is made alkaline to precipitate the non-pyritic iron. Once filtered the sulphate sulphur is precipitated with barium sulphate and determined gravimetrically.

Similarly the pyritic sulphur content is evaluated by desolving ~1g of the coal sample in 15ml hydrochloric acid just below boiling. After the non-pyritic iron is removed the filtered extract is titrated against ammonia to determine the pyritic sulphur content.

The organic sulphur is mathematically determined from the total sulphur minus the sulphate and pyritic sulphur contents.

Ash analysis (BS 1016 part 15)

Coal ash analysis (or ash 11 analysis) is a technique used to evaluate the residue remaining after high temperature combustion under an ambient atmosphere. Coal ash consists mainly of oxides and sulphates the products of the inorganic material oxidation (note that this is not the same as mineral matter i.e. pyrite, quartz, calcite, clays etc, but the high temperature products of these species). The reader is referred to BS1016 part 15 for experimental methods relating to the determination of coal ash components.

Normative analysis

The Normative analysis is an approximate evaluation of the mineral matter quantity and composition of a coal. The evaluation derives the mineral matter from Forms of sulphur and Ash analyses and assumed molecular oxidation formula from the most common associated mineral matter. Pyrite and Gypsum quantities are approximated from a molecular balance based upon the Forms of sulphur analysis whilst the remaining selected minerals are derived from a molecular balance based upon the Ash analysis.

Mineral	**Formula**	**Oxidation product formula**
Pyrite	FeS_2	SO_2, Fe_2O_3
Gypsum	$CaSO_4.2H_2O$	CaO, SO_2, H_2O
Ankerite	$Ca(Fe, Mg, Mn)(CO_3)_2$	Mn_3O_4, Fe_2O_3, MgO, CaO, CO_2
Apatite	$Ca_5(PO_4)_3(F,CL,OH)$	CaO, P_2O_5, H_2O, HCl, HF
Mica	Muscovite-$KAl_2(AlSi_3O_{10})(F,OH)_2$ Biotite-$K(Fe,Mg)_3AlSi_3O_{10}(F,OH)_2$ Lepidolite-$Kli_2Al(Al,Si)_3O_{10}(F,OH)_2$	SiO_2, Al_2O_3, K_2O, HF, H_2O, LiO_2, CaO, $NaOH$, BaO, MgO
Kaolin	Kaolinite-$Al_2Si_2O_5(OH)_4$	Al_2O_3, SiO_2, H_2O
Quartz	SiO_2	SiO_2
Rutile	TiO_2	TiO_2
Siderite	$FeCO_3$	Fe_2O_3, CO_2
Dolomite	$CaMg(CO_3)_2$	CaO, MgO, CO_2
Calcite	$CaCO_3$	CaO, CO_2

The molecular phase change balance (Ash analysis) is assumed as the most likely original state prior to total combustion.

The number of moles of each mineral have been derived from the following assumptions:

Pyrite = Number of moles of pyritic sulphur/2
Gypsum = Number of moles of sulphate sulphur
Ankerite = Number of moles of Mn_3O_4 × 28.6
Apatite = Number of moles of $P_2O_5 \times 2/3$
Mica = Number of moles of K_2O
Kaolin = Number of moles of Al_2O_3 – number of moles of mica
Quartz = Number of moles of SiO_2 – number of moles of (mica × 6) – number of moles of (kaolin × 2)
Rutile = Number of moles of TiO_2
Siderite = Number of moles of (2 × Fe_2O_3) – number of moles of (pyrite) – number of moles of (ankerite)
Dolomite = Number of moles of remaining from MgO after ankerite
Calcite = Number of moles of remaining after apatite, gypsum, dolomite and ankerite

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APPENDIX B

ANALYSIS OF REFERENCE COAL

Proximate analysis (as-received basis unless stated in accord with BS1016) of group 1 coals (University of Birmingham)

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{\mathrm{dmmf}}$ %
F-1	1.6 (0.08)	1.8 (0.03)	4.6 (0.30)	92.0	4.8
F-2	0.8 (0.06)	4.0 (0.01)	12.7 (0.74)	82.5	13.4
F-3	1.0 (0.02)	3.8 (0.04)	28.5 (0.65)	67.7	29.8
F-4	5.8 (0.03)	5.2 (0.07)	33.0 (1.65)	56.0	37.6
F-5	6.1 (0.06)	3.4 (0.05)	34.8 (0.24)	55.7	38.7
F-6	6.1 (0.09)	4.4 (0.13)	35.7 (0.40)	53.8	40.4
F-7	4.7 (0.04)	5.0 (0.02)	37.1 (0.54)	53.2	41.6
F-8	13.3 (0.02)	7.4 (0.07)	34.9 (0.07)	44.4	45.1

Mean value given from 5 trials, Standard deviations given in brackets

Proximate analysis (as-received basis unless stated in accord with BS1016) of group 1 coals (CRE Group)

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{\mathrm{dmmf}}$ %
F-1	1.6	1.8	4.6	92.0	4.8
F-2	0.8	4.0	12.7	82.5	13.4
F-3	1.0	2.8	28.5	67.7	29.8
F-4	5.6	5.2	33.0	56.0	37.6
F-5	6.1	3.4	34.8	55.7	38.7
F-6	6.1	4.4	35.7	53.8	40.4
F-7	4.7	5.0	37.1	53.2	41.6
F-8	13.3	7.4	34.9	44.4	45.1

Ultimate analysis (dry, mineral matter free basis) of group 1 coals (CRE Group)

Coal sample	Sulphur %	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
F-1	0.57	90.10	2.43	1.10	5.80
F-2	0.66	88.40	3.66	1.50	5.78
F-3	1.06	83.50	4.60	1.30	9.54
F-4	2.12	73.80	4.56	1.80	17.72
F-5	1.70	74.80	4.43	1.70	17.37
F-6	1.43	71.70	4.30	1.30	21.27
F-7	1.39	76.00	4.96	1.90	15.75
F-8	1.98	63.50	3.95	1.50	29.07

Ash analysis (percentage based on ash content) of group 1 coals (CRE Group)

Coal	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
Na_2O %	3.7	2.9	0.6	2.8	6.1	1.5	4.2	0.3
K_2O %	0.3	0.4	0.4	1.3	1.4	0.5	1.5	2.4
CaO %	3.6	3.9	2.7	2.3	5.0	12.0	12.5	8.7
MgO %	2.0	1.0	0.4	0.8	0.7	2.5	0.6	2.8
Fe_2O_3 %	15.1	3.7	3.5	25.1	16.5	11.2	23.2	27.5
Al_2O_3 %	31.0	39.5	38.0	26.5	29.1	23.9	17.6	17.1
SiO_2 %	39.1	39.2	36.3	39.3	39.3	36.8	31.4	31.5
SO_2 %	3.4	2.6	0.8	2.1	3.4	12.9	2.6	7.1
TiO_2 %	0.4	0.6	1.2	0.9	1.3	1.1	0.6	0.6
Mn_2O_4 %	0.2	0.1	0.1	0.1	0.1	0.4	1.1	0.4
P_2O_5 %	0.2	5.2	10.2	0.2	0.3	0.3	6.6	0.8

Forms of sulphur (dry basis) of group 1 coals (CRE Group)

Coal	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
SO_4	0.02	0.02	0	<0.05	<0.1	<0.1	<0.1	0.08
Pyritic	0.02	0.03	0.03	1.1	0.4	0.28	0.73	1.34
Organic	0.59	0.67	0.84	1.11	1.1	1.12	0.84	0.82
Total	0.63	0.72	0.87	2.21	1.5	1.4	1.57	2.24

Normative analysis (percentage based on mineral matter content) of group 1 coals

Coal	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
Pyrite	1.7	1.1	1.5	1.5	16.6	9.5	14.9	27.0
Gypsum	1.6	0.7	0.0	0.0	4.0	3.2	2.0	1.5
Ankerite	10.0	2.4	4.8	4.8	4.8	20.5	38.6	20.5
Dolomite	3.5	2.6	0.0	0.0	0.7	1.8	0.0	2.9
Apatite	0.4	9.3	18.1	18.1	0.5	0.6	8.5	1.5
Calcite	0.0	0.0	0.0	0.0	1.6	5.7	0.0	0.5
Mica	2.0	2.6	2.6	2.6	9.0	3.4	7.0	16.3
Kaolin	58.8	73.3	69.8	69.8	46.7	44.8	17.6	18.7
Quartz	2.0	0.0	0.0	0.0	3.8	6.9	5.8	9.1
Rutile	0.3	0.5	0.9	0.9	1.0	0.9	0.3	0.5
Siderite	13.1	2.5	1.3	1.3	0.9	0.0	0.0	1.0
Na_2SO_4	6.6	5.0	1.0	1.0	10.5	2.7	5.3	0.5
Mineral content %	2.0	4.6	3.2	6.6	4.3	5.6	6.1	9.6

Mineral matter content given as a percentage of the coal content

Proximate analysis (as-received basis unless stated in accord with BS1016) of group 2 coals (University of Birmingham)

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
P-1	3.73 (0.15)	18.20 (0.02)	29.69 (0.23)	48.39	38.03
P-2	4.42 (0.09)	13.45 (0.06)	32.25 (0.20)	49.88	39.27
P-3	4.31 (0.09)	19.20 (0.01)	30.29 (0.23)	46.20	39.60
P-4	2.26 (0.17)	7.70 (0.54)	34.45 (0.23)	55.60	38.26
P-5	4.09 (0.06)	13.54 (0.01)	34.08 (0.12)	48.29	41.37
P-6	4.41 (0.17)	12.91 (0.06)	30.33 (0.32)	52.35	36.68
P-7	3.56 (0.08)	6.10 (0.02)	34.02 (0.13)	56.32	37.66
P-8	3.17 (0.05)	21.87 (1.31)	26.72 (0.36)	48.24	35.65

Mean value given from 5 trials, Standard deviations given in brackets

Proximate analysis (as-received basis unless stated in accord with BS1016) of group 2 coals (CRE Group)

Coal sample	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
P-1	3.7	18.7	29.8	47.8	38.4
P-2	5.2	13.5	32.5	48.8	39.9
P-3	4.7	16.3	30.7	48.3	38.8
P-4	1.9	19.0	29.7	49.4	37.5
P-5	2.3	15.7	30.2	51.8	36.8
P-6	5.0	14.8	32.7	47.6	40.7
P-7	-	-	-	-	-
P-8	3.8	20.4	28.3	47.5	38.7

Ultimate analysis (dry, mineral matter free basis) of group 2 coals (CRE Group)

Coal sample	Sulphur %	Carbon %	Hydrogen %	Nitrogen %	Oxygen %
P-1	1.95	65.70	4.13	1.44	26.19
P-2	1.68	69.10	4.34	0.86	23.17
P-3	1.83	68.30	4.16	1.14	24.02
P-4	2.82	67.70	4.26	1.53	23.11
P-5	2.18	67.70	4.65	1.30	24.02
P-6	1.55	67.70	3.93	1.19	24.34
P-7	-	-	-	-	-
P-8	1.33	84.50	4.90	1.80	6.5

Ash analysis (percentage based on ash content) of group 2 coals (CRE Group)

Coal	P-1	P-2	P-3	P-4	P-5	P-6	P-7	P-8
Na_2O %	1.1	2.2	0.8	0.7	0.4	0.4	-	1.6
K_2O %	3.6	2.9	2.6	3.2	2.0	3.0	-	4.0
CaO %	2.1	2.9	2.6	1.0	7.4	1.8	-	2.5
MgO %	1.5	1.5	1.6	1.2	2.0	0.9	-	1.6
Fe_2O_3 %	10.8	10.4	12.9	16.9	9.3	12.1	-	7.2
Al_2O_3 %	26.7	24.2	24.1	23.5	21.0	26.1	-	24.9
SiO_2 %	51.4	52.7	52.9	51.9	50.9	54.0	-	55.4
SO_2 %	1.3	1.9	1.5	0.2	0.7	1.3	-	2.1
TiO_2 %	0.8	0.8	0.1	0.1	0.2	0.1	-	0.9
Mn_2O_4 %	0.1	0.1	0.1	0.1	0.2	0.1	-	0.1
P_2O_5 %	0.3	0.2	0.5	0.4	0.7	0.3	-	0.2

Forms of sulphur (dry basis) of group 2 coals (CRE Group)

Coal	P-1	P-2	P-3	P-4	P-5	P-6	P-7	P-8
SO_4	0.18	0.05	0.12	0.24	0.06	0.40	0.29	0.10
Pyritic	0.67	0.52	0.67	1.14	0.52	2.04	0.66	0.56
Organic	1.06	0.90	0.91	1.24	1.23	0.97	1.25	0.76
Total	1.91	1.47	1.70	2.62	1.81	3.41	2.20	1.42

Normative analysis (percentage based on mineral matter content) of group 2 coals

Coal	P-1	P-2	P-3	P-4	P-5	P-6	P-7	P-8
Pyrite	5.6	6.1	6.4	9.1	5.7	18.7	-	3.9
Gypsum	1.4	0.6	1.1	1.8	0.11	3.5	-	5.1
Ankerite	5.4	5.4	5.3	5.2	11.1	4.9	-	4.9
Dolomite	3.8	3.9	4.2	2.6	3.9	1.4	-	3.8
Apatite	0.6	0.4	1.0	0.8	1.4	0.5	-	0.4
Calcite	0.0	0.0	0.0	0.0	3.2	0.0	-	0.0
Mica	25.6	20.9	18.4	22.1	14.7	19.6	-	26.1
Kaolin	31.7	31.6	32.9	26.9	31.6	31.7	-	23.0
Quartz	16.7	20.5	20.4	19.7	22.6	17.9	-	20.0
Rutile	0.7	0.7	0.7	0.6	0.7	1.0	-	6.9
Siderite	6.4	5.6	8.2	9.9	3.6	0.0	-	3.1
Na_2SO_4	2.1	4.3	1.5	1.3	0.14	0.7	-	2.8
Mineral content %	21.5	15.5	18.8	22.2	17.7	18.8	-	23.2

Mineral matter content given as a percentage of the coal content

Calorific value (dry, mineral free basis) and Hardgrove Grindability Index of group 1 coals

Coal	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
Calorific Value (MJ/kg)	36.00	36.79	36.45	34.52	34.44	32.57	34.98	31.72
HGI	23	73	85	56	50	44	42	43

Calorific value (dry, mineral free basis) and Hardgrove Grindability Index of group 2 coals

Coal	P-1	P-2	P-3	P-4	P-5	P-6	P-7	P-8
Calorific Value (MJ/kg)	34.45	34.42	34.72	35.30	34.10	35.80	36.21	33.80
HGI	65	53	59	64	58	59	-	58

As-received bulk size distribution of group 1 coal (wieght %)

Size range	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
>16.0 mm	97.4	99.2	95.2	42.3	96.3	29.7	95.8	26.4
16.0-13.2 mm	0.6	0.0	0.3	8.7	0.6	18.9	0.7	6.6
13.2-11.2 mm	0.4	0.0	0.2	7.6	0.5	16.9	0.9	7.6
11.2-8.0 mm	0.6	0.0	0.5	13.5	0.7	17.4	1.0	14.3
8.0-5.6 mm	0.4	0.1	0.5	8.1	0.4	6.8	0.5	10.7
5.6-4.0 mm	0.2	0.1	0.5	3.8	0.2	2.7	0.2	7.0
4.0-2.8 mm	0.1	0.1	0.5	2.2	0.2	1.6	0.2	6.0
2.8-2.0 mm	0.1	0.1	0.4	1.4	0.2	0.9	0.1	4.3
2.0-1.4 mm	0.1	0.1	0.4	1.2	0.2	0.6	0.1	3.7
1.4-1.0 mm	0.0	0.1	0.2	0.7	0.1	0.4	0.1	2.4
1.0-0.71 mm	0.0	0.1	0.2	0.7	0.1	0.4	0.1	2.1
710-500 μm	0.0	0.1	0.3	0.5	0.1	0.2	0.1	1.8
500-355 μm	0.0	0.0	0.1	0.6	0.1	0.3	0.0	1.2
355-250 μm	0.0	0.0	0.2	0.7	0.1	0.3	0.1	1.1
250-180 μm	0.0	0.0	0.1	0.8	0.1	0.3	0.1	0.9
180-125 μm	0.0	0.0	0.1	1.0	0.0	0.4	0.1	0.7
125-90 μm	0.0	0.0	0.0	1.0	0.0	0.4	0.1	0.6
90-75 μm	0.0	0.0	0.0	1.2	0.0	0.4	0.0	0.5
75-63 μm	0.0	0.0	0.0	0.7	0.0	0.6	0.0	0.4
63-45 μm	0.0	0.0	0.0	2.0	0.0	0.4	0.0	0.6
45-38 μm	0.0	0.0	0.0	0.7	0.0	0.2	0.0	0.6
<38 μm	0.0	0.0	0.0	0.6	0.0	0.1	0.0	0.5
Total	100	100	100	100	100	100	100	100

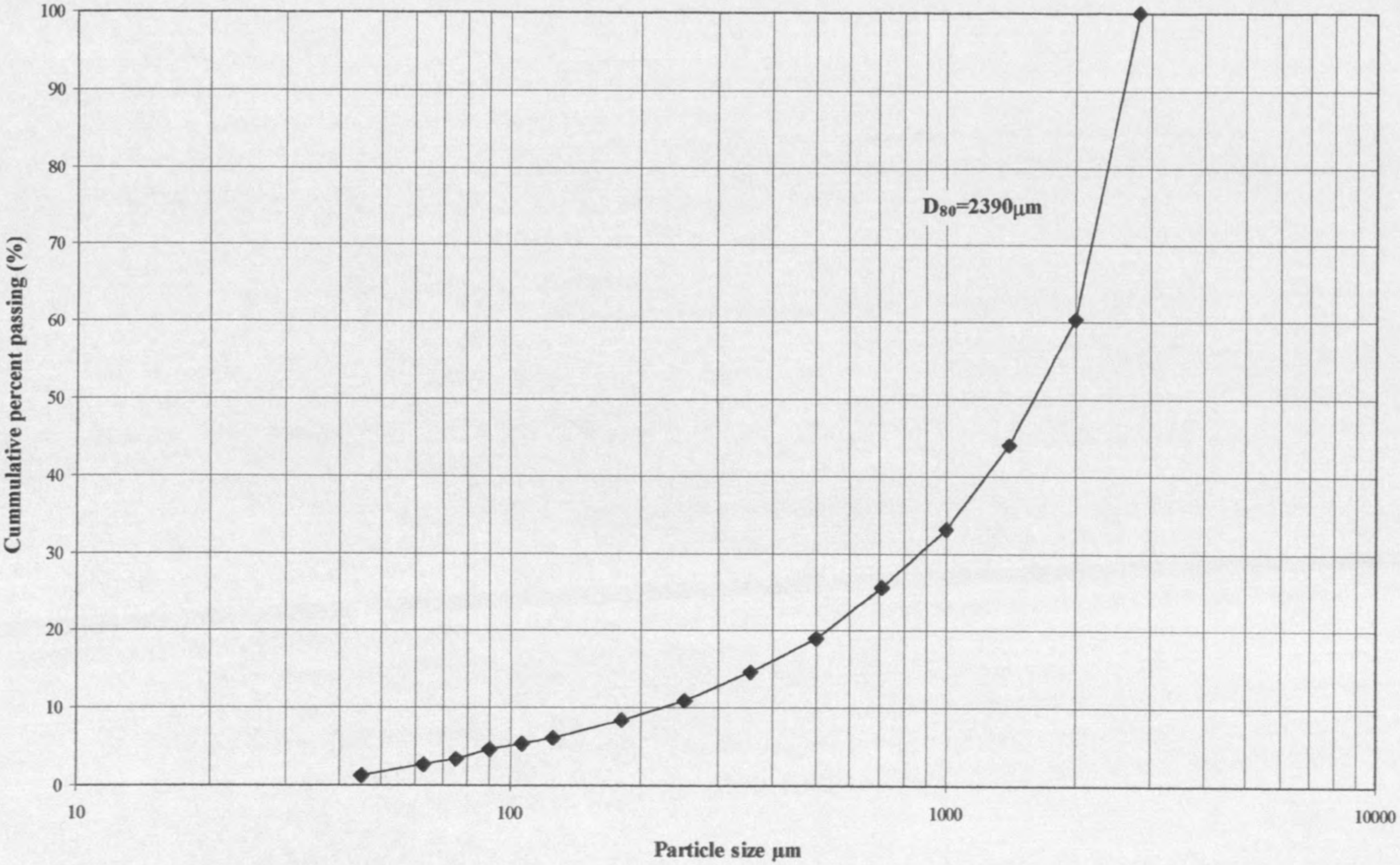
Jaw Crusher product size distribution of group 1 coal (wieght %)

Size range	F-1	F-2	F-3	F-4	F-5	F-6	F-7	F-8
>16.0 mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
16.0-13.2 mm	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
13.2-11.2 mm	22.8	5.9	6.2	15.6	9.6	22.9	13.1	15.7
11.2-8.0 mm	32.2	17.4	20.8	26.6	23.8	31.1	26.5	23.4
8.0-5.6 mm	16.4	15.7	17.0	17.9	18.2	16.6	18.1	16.5
5.6-4.0 mm	9.0	11.5	11.8	9.1	11.3	8.3	11.2	9.9
4.0-2.8 mm	6.0	10.7	10.5	6.1	9.5	5.4	8.0	8.1
2.8-2.0 mm	4.1	9.1	8.6	4.5	7.0	3.7	5.6	5.7
2.0-1.4 mm	3.0	7.9	6.7	3.4	5.6	2.8	5.0	4.8
1.4-1.0 mm	1.9	6.0	4.8	2.0	3.5	1.6	2.9	3.0
1.0-0.71 mm	1.3	4.4	3.7	1.9	3.1	1.4	2.5	2.6
710-500 µm	1.2	4.2	3.2	1.7	2.6	1.2	2.2	2.3
500-355 µm	0.7	2.1	1.8	1.1	1.4	0.7	1.3	1.5
355-250 µm	0.4	1.7	1.5	1.1	1.2	0.7	1.0	1.2
250-180 µm	0.3	1.0	1.1	1.1	0.9	0.6	0.7	1.0
180-125 µm	0.3	0.9	0.6	1.2	0.7	0.6	0.7	0.8
125-90 µm	0.1	0.4	0.5	1.2	0.4	0.5	0.5	0.7
90-75 µm	0.1	0.3	0.3	1.2	0.2	0.4	0.2	0.5
75-63 µm	0.0	0.1	0.2	0.9	0.2	0.7	0.1	0.4
63-45 µm	0.0	0.2	0.3	2.1	0.3	0.5	0.2	0.7
45-38 µm	0.0	0.3	0.3	0.8	0.4	0.2	0.3	0.6
<38 µm	0.1	0.2	0.1	0.6	0.1	0.1	0.2	0.5
Total	100	100	100	100	100	100	100	100

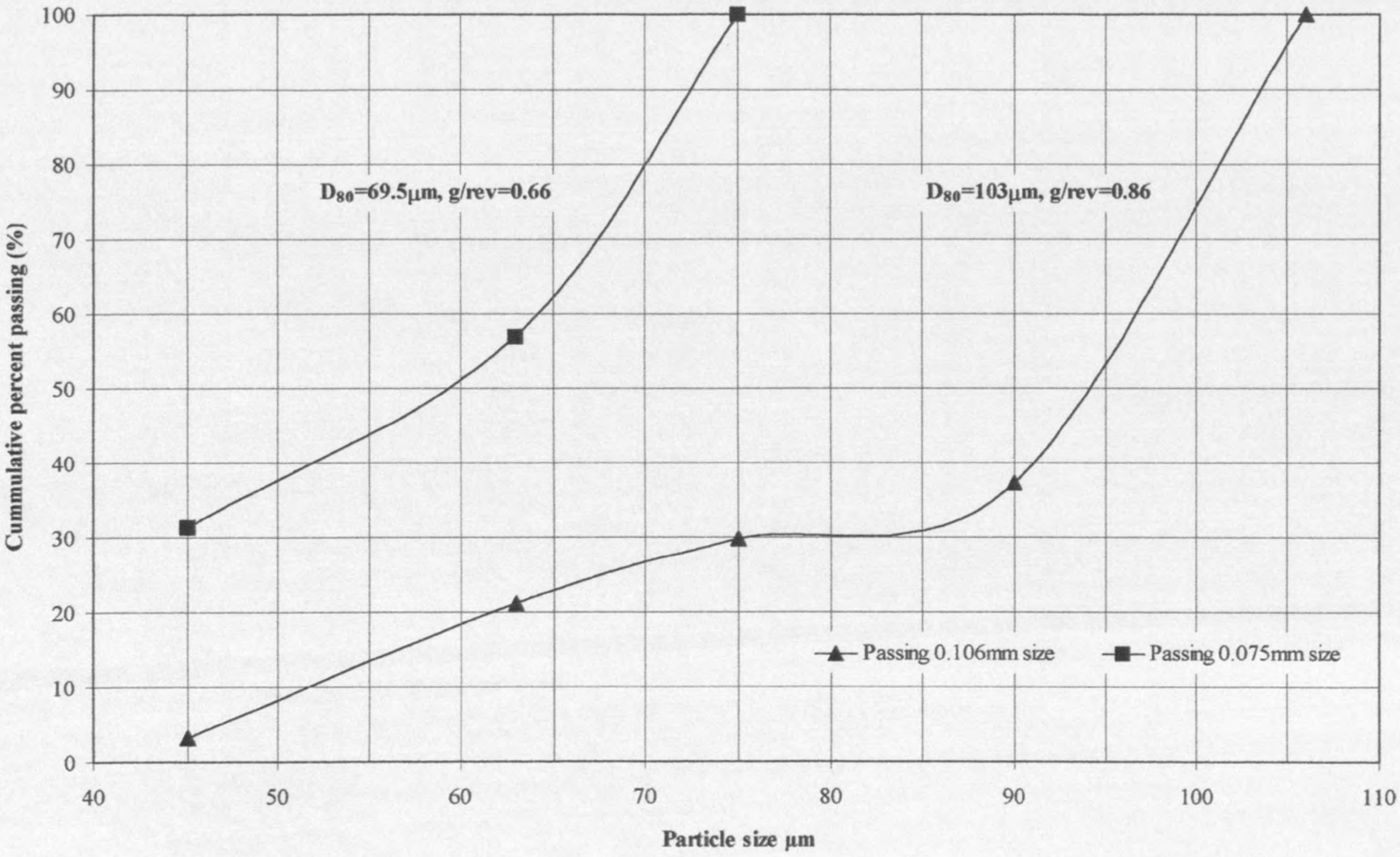
As-received bulk size distribution of group 2 coal (wieght %)

Size range	P-1	P-2	P-3	P-4	P-5	P-6	P-7
>32.0 mm	0.00	0.34	0.00	0.00	0.00	1.32	0.00
32.0-22.0 mm	0.16	0.15	1.51	31.51	0.00	0.63	0.00
22.0-16.0 mm	8.60	19.51	16.10	54.95	1.80	16.03	54.66
16.0-11.3 mm	7.91	12.98	14.91	6.10	15.63	12.33	23.88
11.3-8.0 mm	10.11	11.96	16.29	1.94	36.22	12.82	6.98
8.0-5.6 mm	10.58	11.02	12.26	0.98	21.02	11.94	3.69
5.6-4.0 mm	10.03	8.65	7.78	0.70	5.33	11.81	2.41
4.0-2.8 mm	10.58	7.93	6.09	0.56	3.44	10.49	1.91
2.8-2.0 mm	8.93	6.06	4.29	0.44	2.52	14.57	1.29
2.0-1.4 mm	8.50	4.96	3.86	0.45	2.72	5.85	1.10
1.4-1.0 mm	4.56	3.18	2.49	0.32	1.49	1.70	0.58
1.0-0.71 mm	4.69	2.78	3.34	0.31	1.60	0.30	0.58
710-500 μm	3.40	2.32	2.00	0.27	1.29	0.18	0.54
500-355 μm	2.73	1.79	1.61	0.24	1.16	0.03	0.48
355-250 μm	2.49	1.71	1.54	0.26	1.24	0.00	0.91
250-180 μm	1.91	1.32	1.58	0.24	1.02	0.00	0.60
180-125 μm	1.87	1.05	1.58	0.21	0.93	0.00	0.29
125-90 μm	1.55	1.17	1.59	0.31	1.29	0.00	0.07
90-63 μm	0.49	0.59	1.11	0.18	0.36	0.00	0.02
63-38 μm	0.85	0.49	0.07	0.03	0.86	0.00	0.01
<38 μm	0.06	0.04	0.00	0.00	0.08	0.00	0.00
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00

Bond test on P-2 coal, Jaw Crusher (-2.8mm) product size distribution



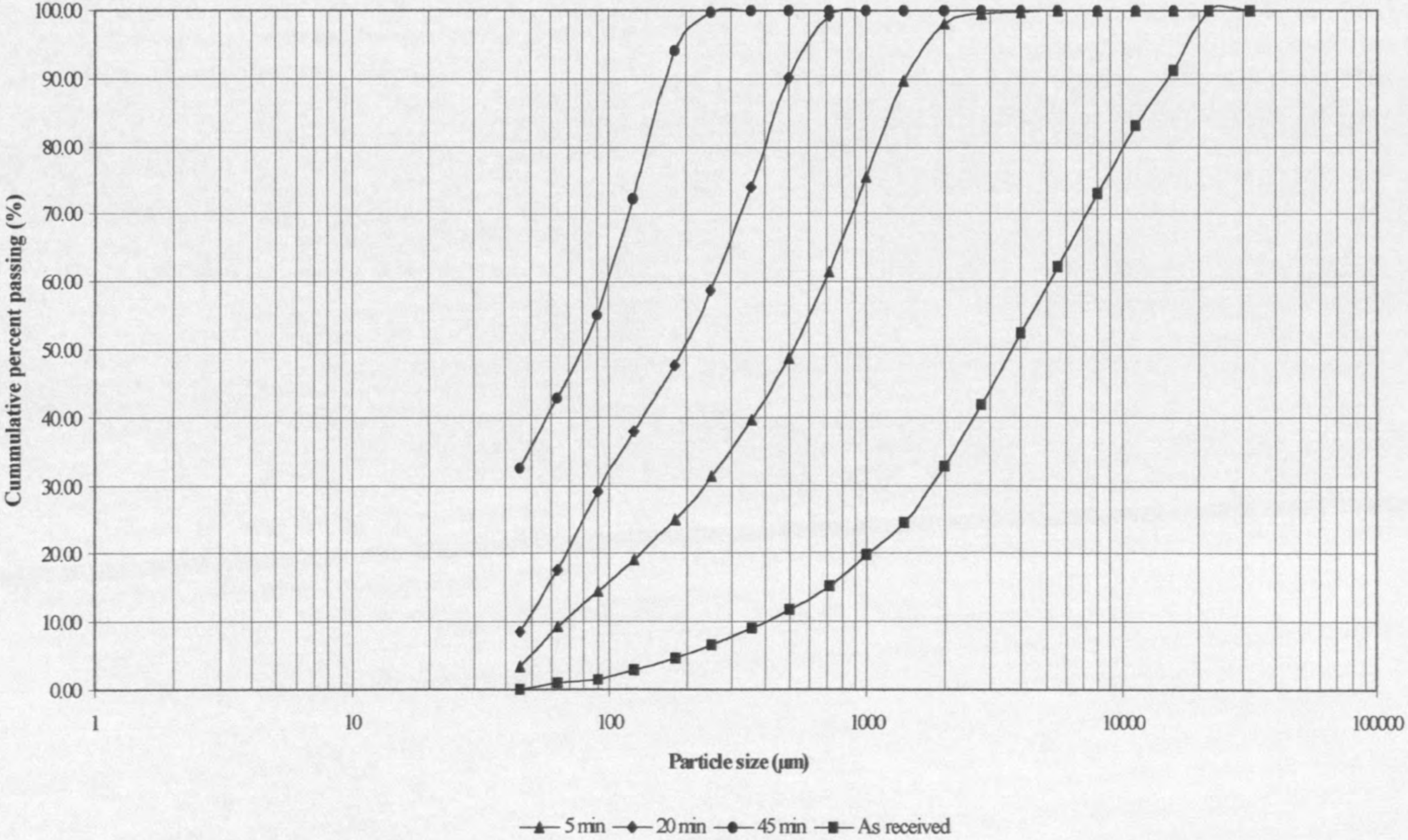
Bond test on P-2 coal, equilibrium condition product undersize



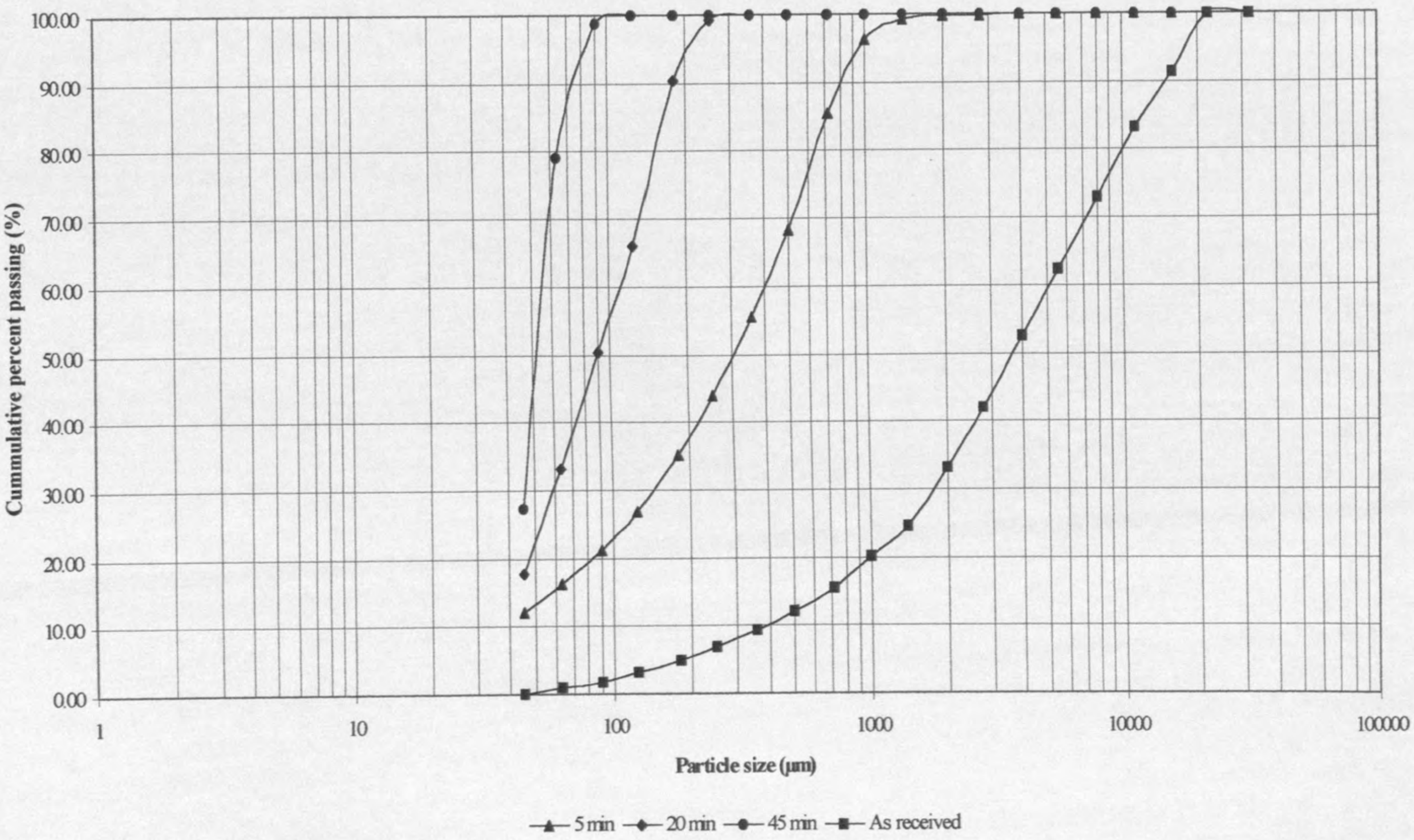
APPENDIX C

ROD MILL OPTIMISATION

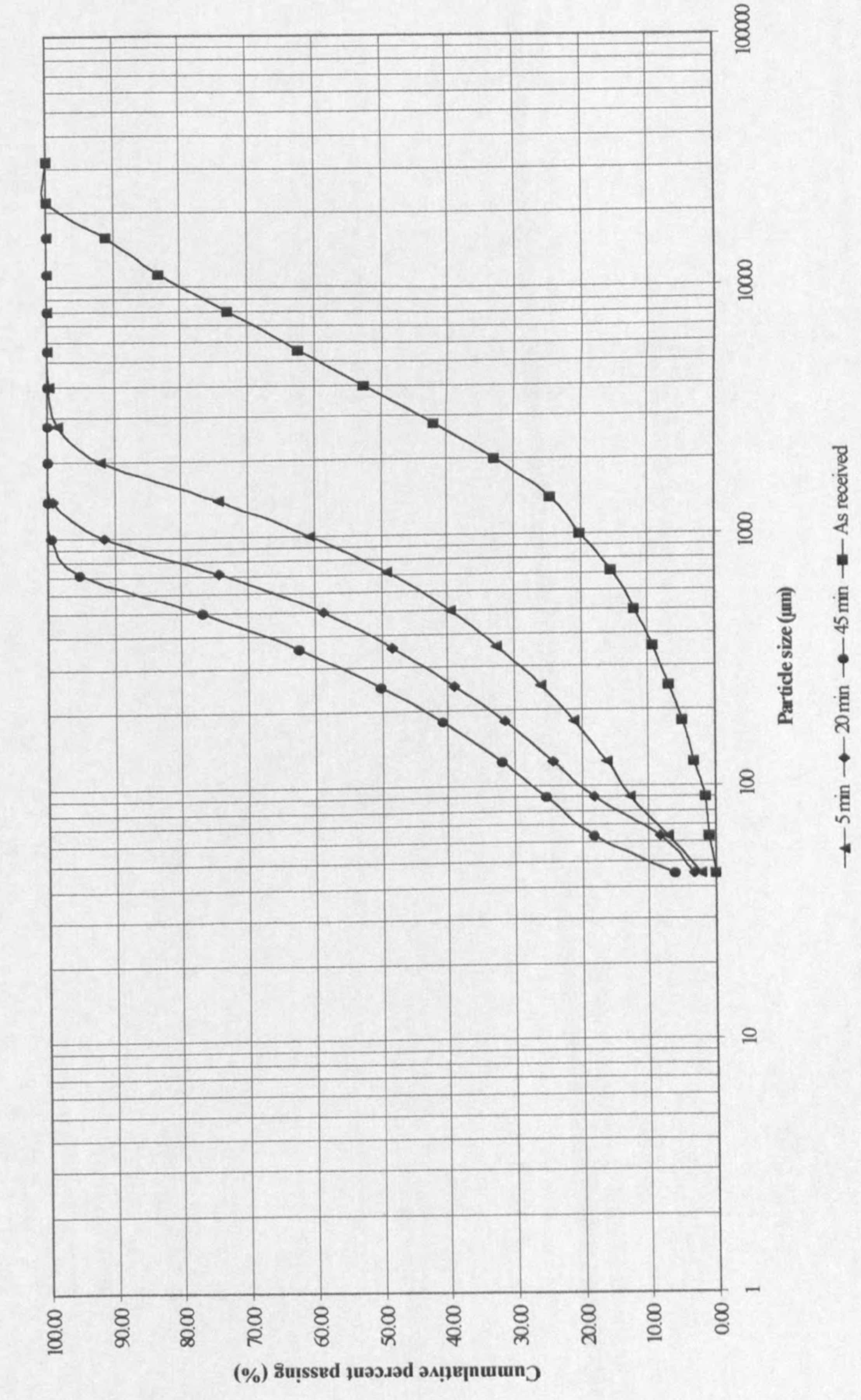
P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-500g
As-received coal



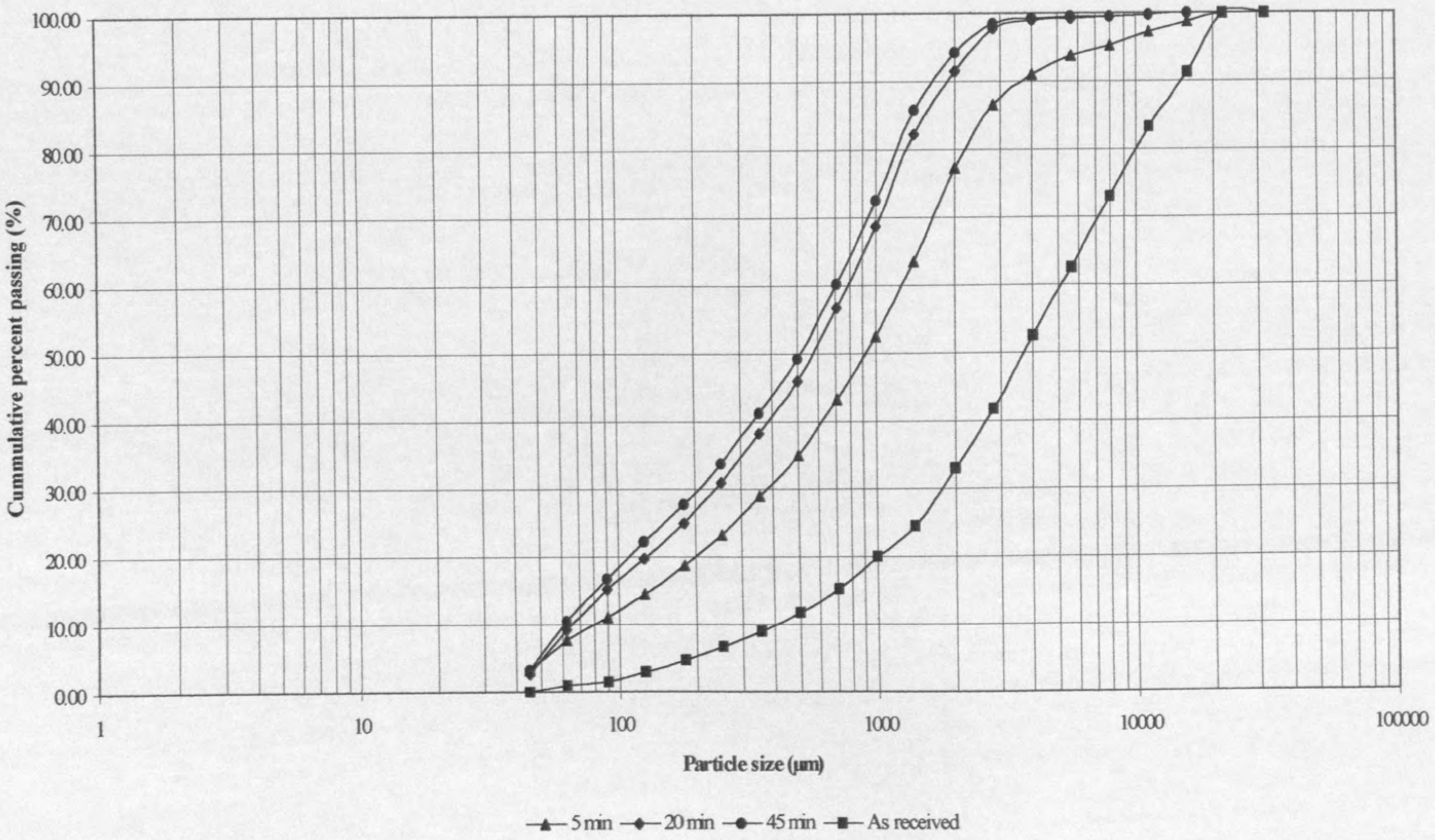
P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-250g
As-received coal

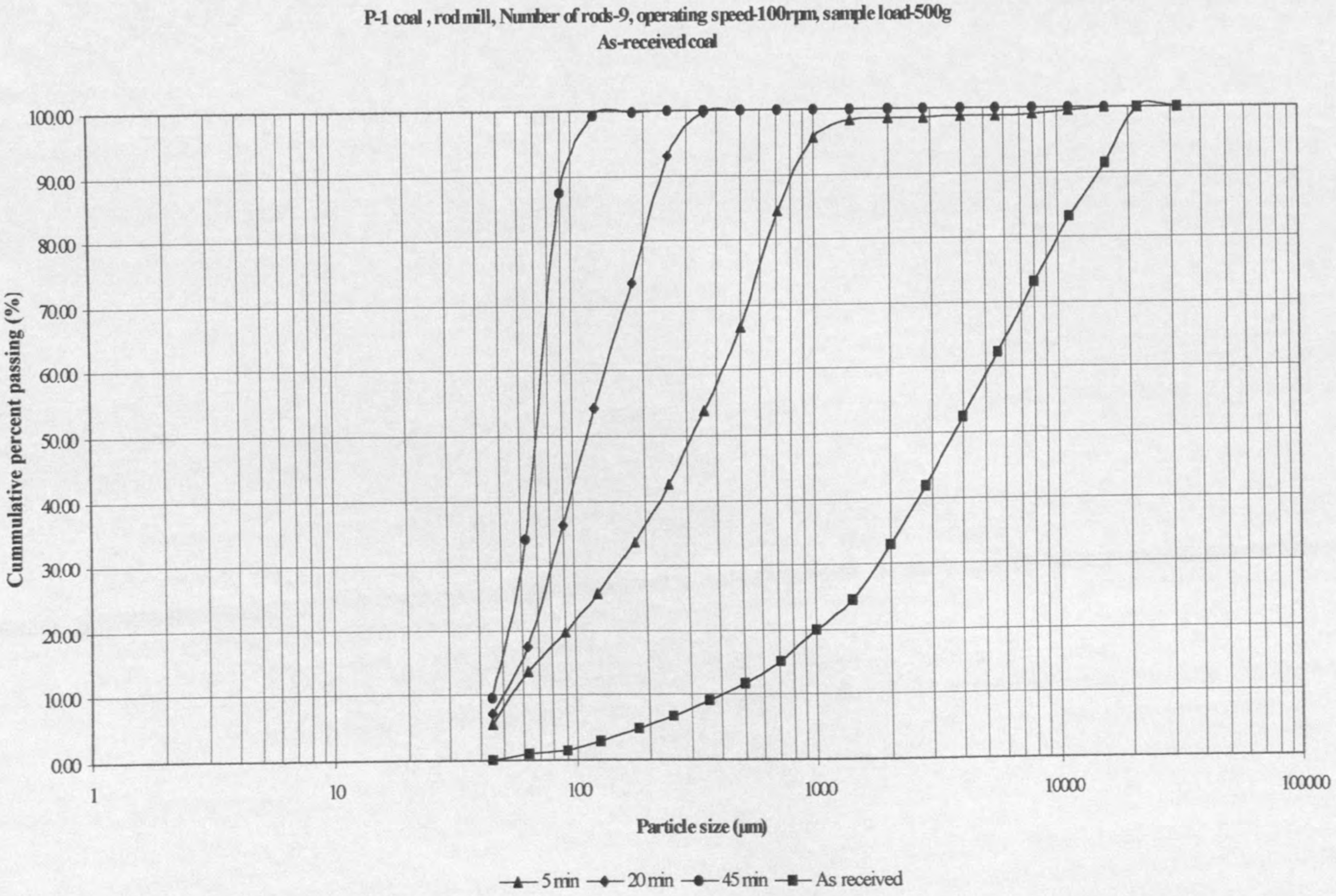


P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-1000g
As-received coal

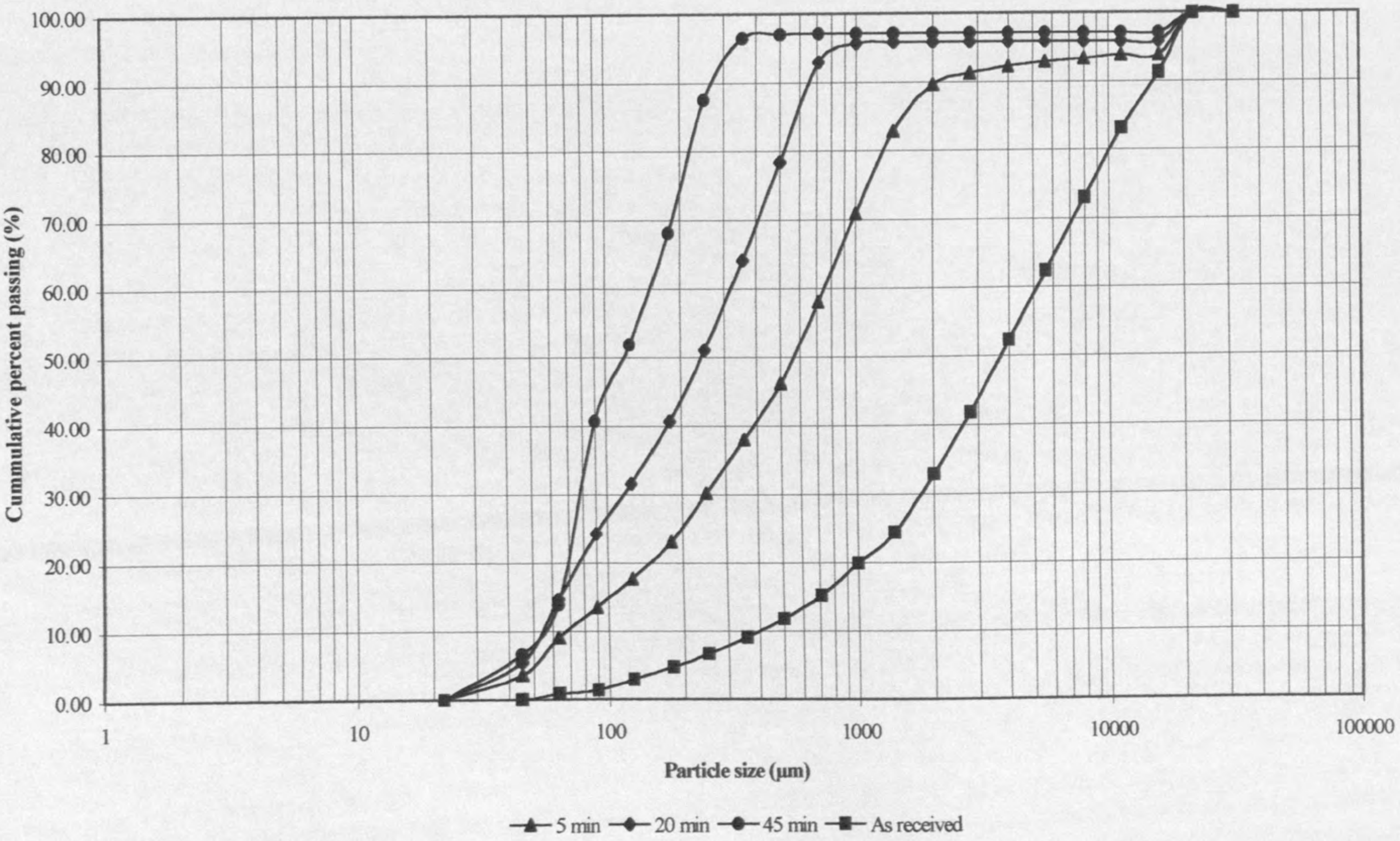


P-1 coal , rod mill, Number of rods-5, operating speed-100rpm, sample load-500g
As-received coal

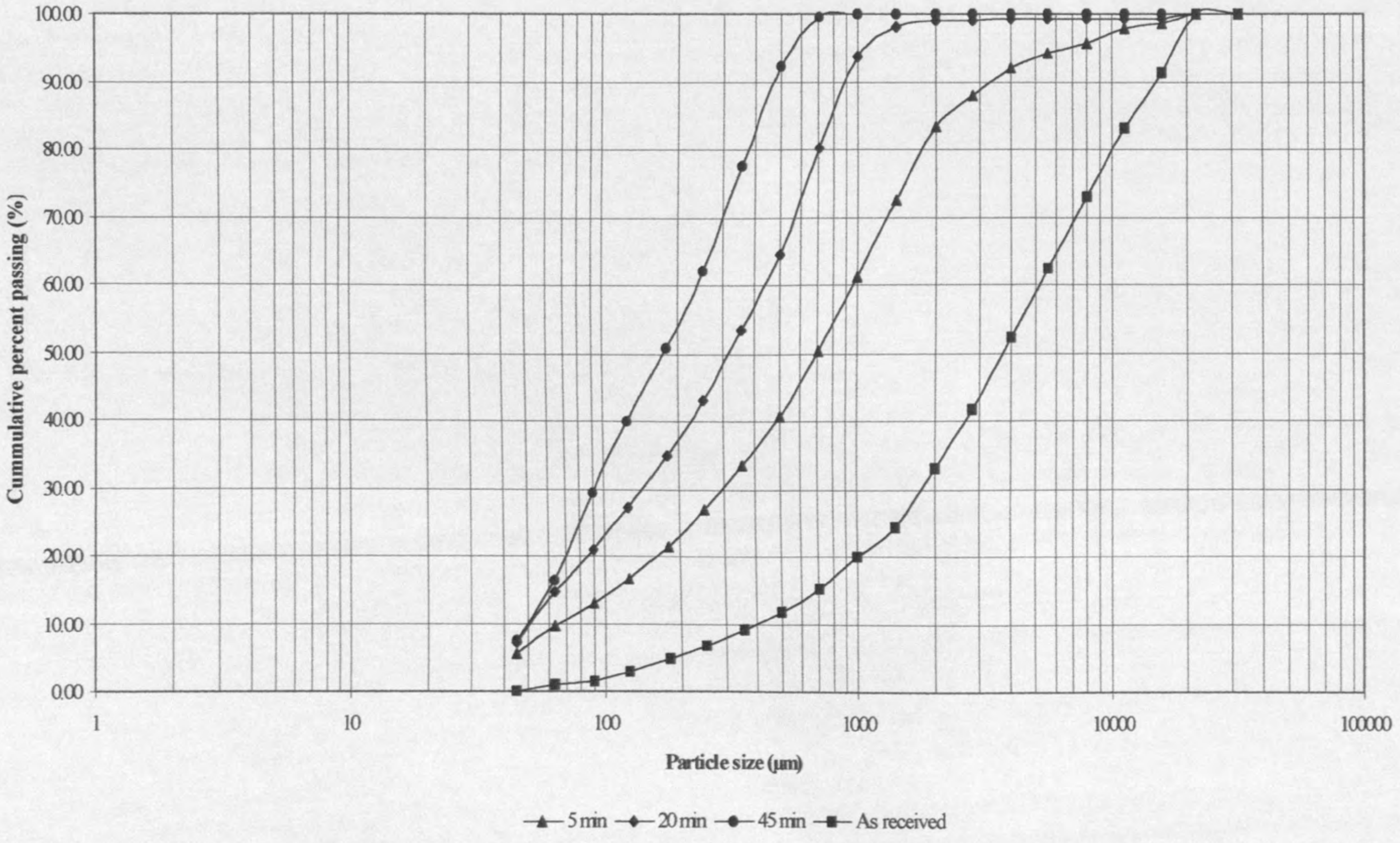



P-1 coal , rod mill, Number of rods-9, operating speed-100rpm, sample load-500g
As-received coal
Cummulative percent passing (%)
100.00
90.00
80.00
70.00
60.00
50.00
40.00
30.00
20.00
10.00
0.00
1
10
100
1000
10000
100000
Particle size (µm)
5 min
20 min
45 min
As received

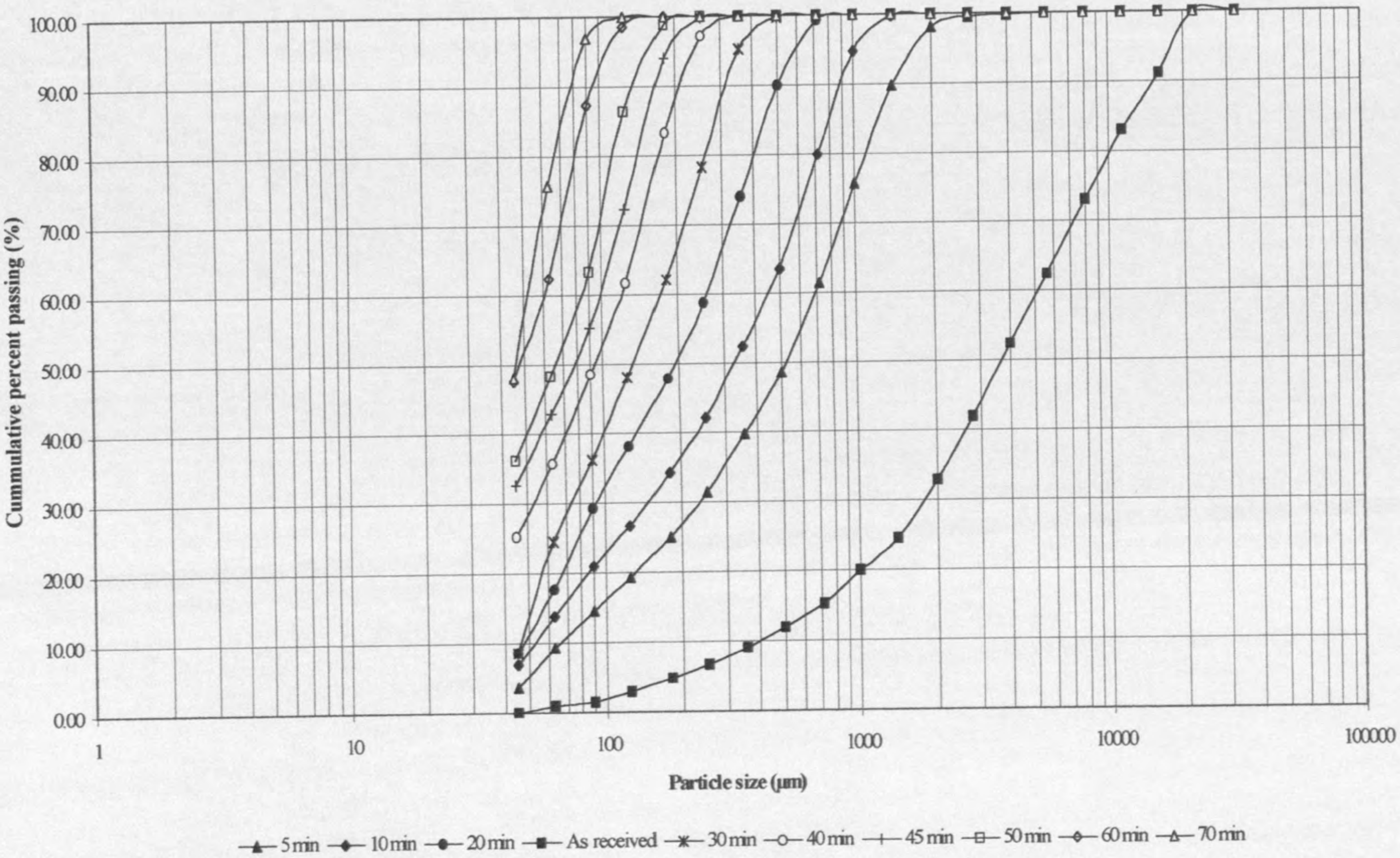
P-1 coal , rod mill, Number of rods-7, operating speed-120rpm, sample load-500g
As-received coal



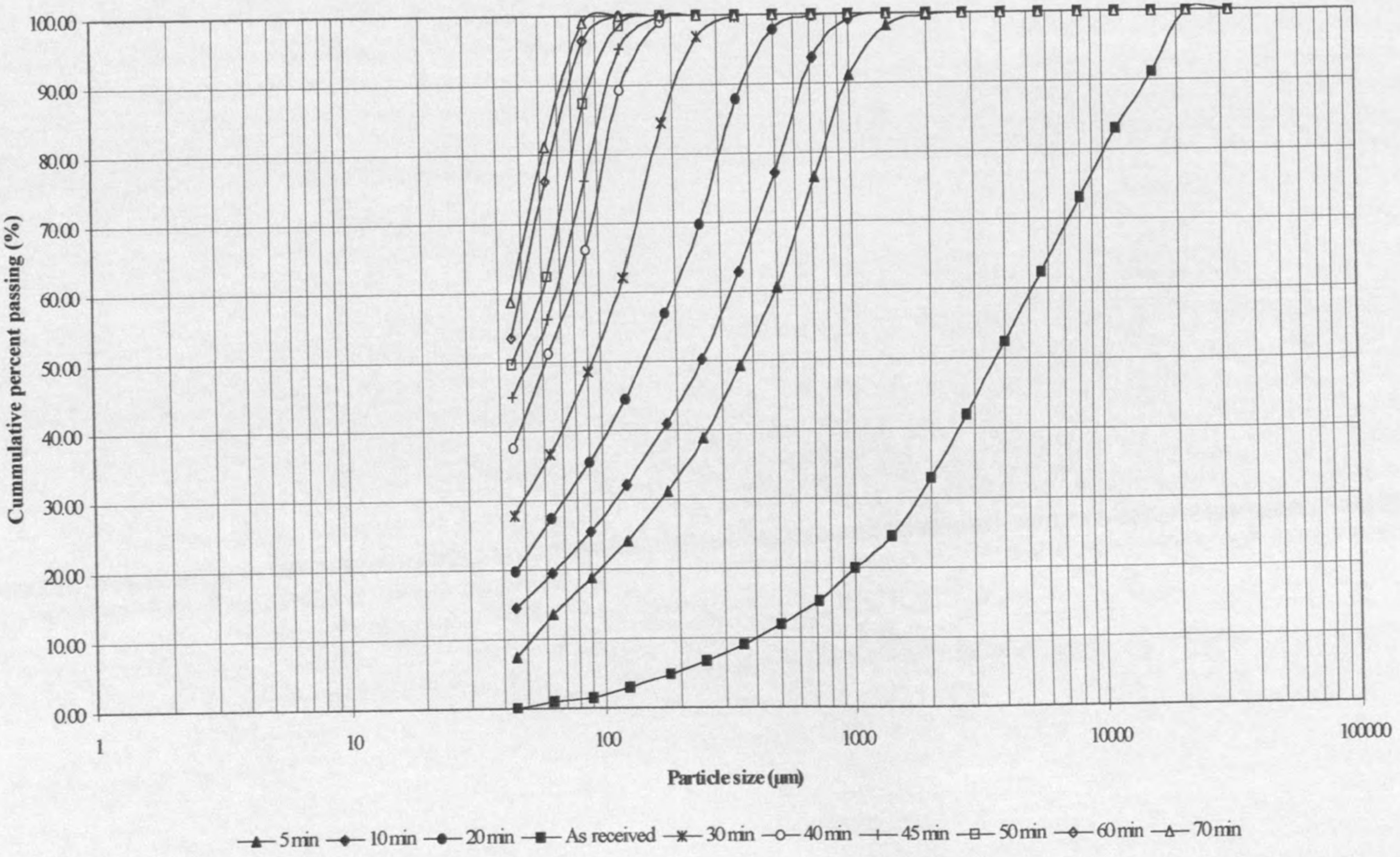
P-1 coal , rod mill, Number of rods-7, operating speed-60rpm, sample load-500g
As-received coal



P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-500g
As-received coal



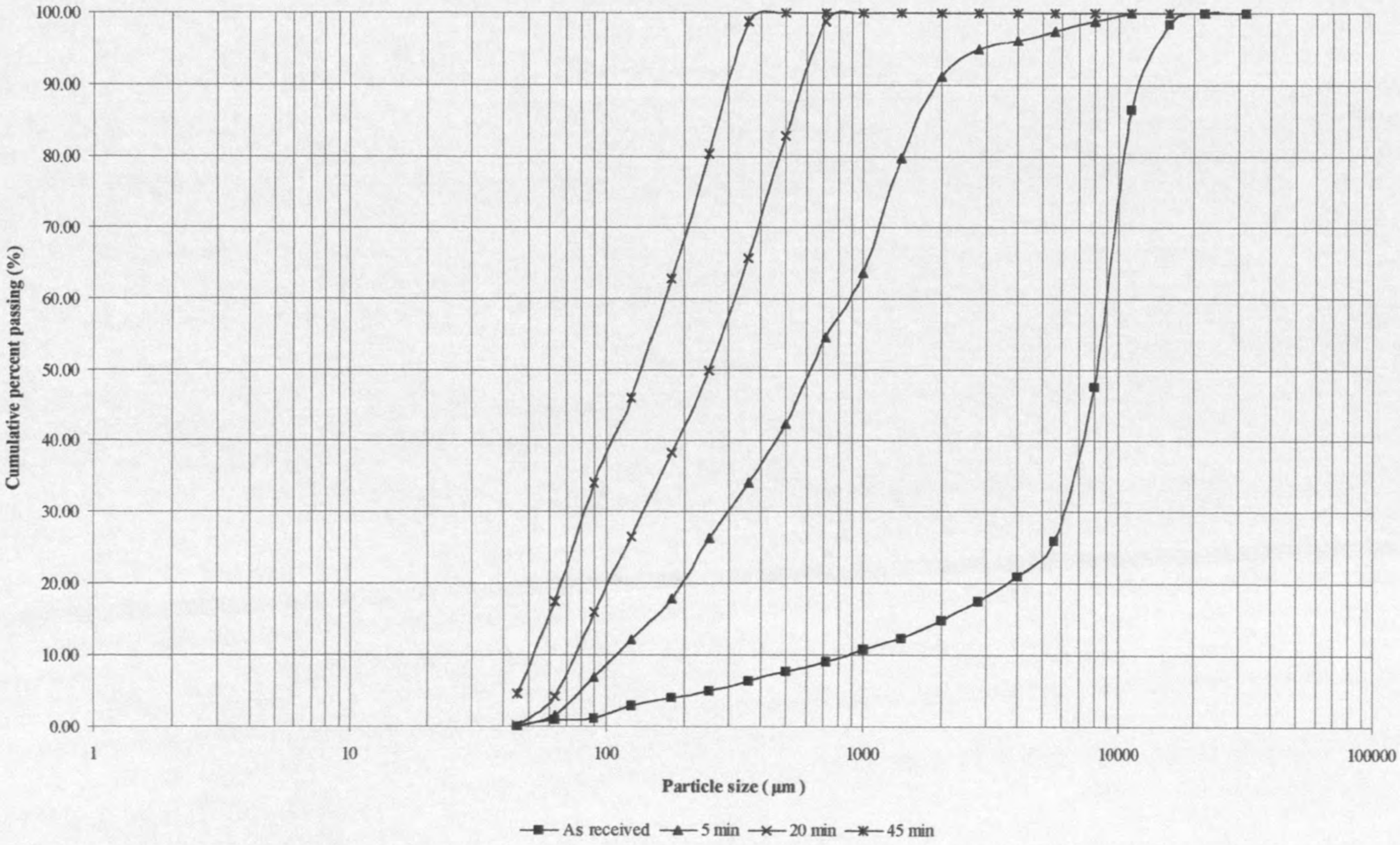
P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



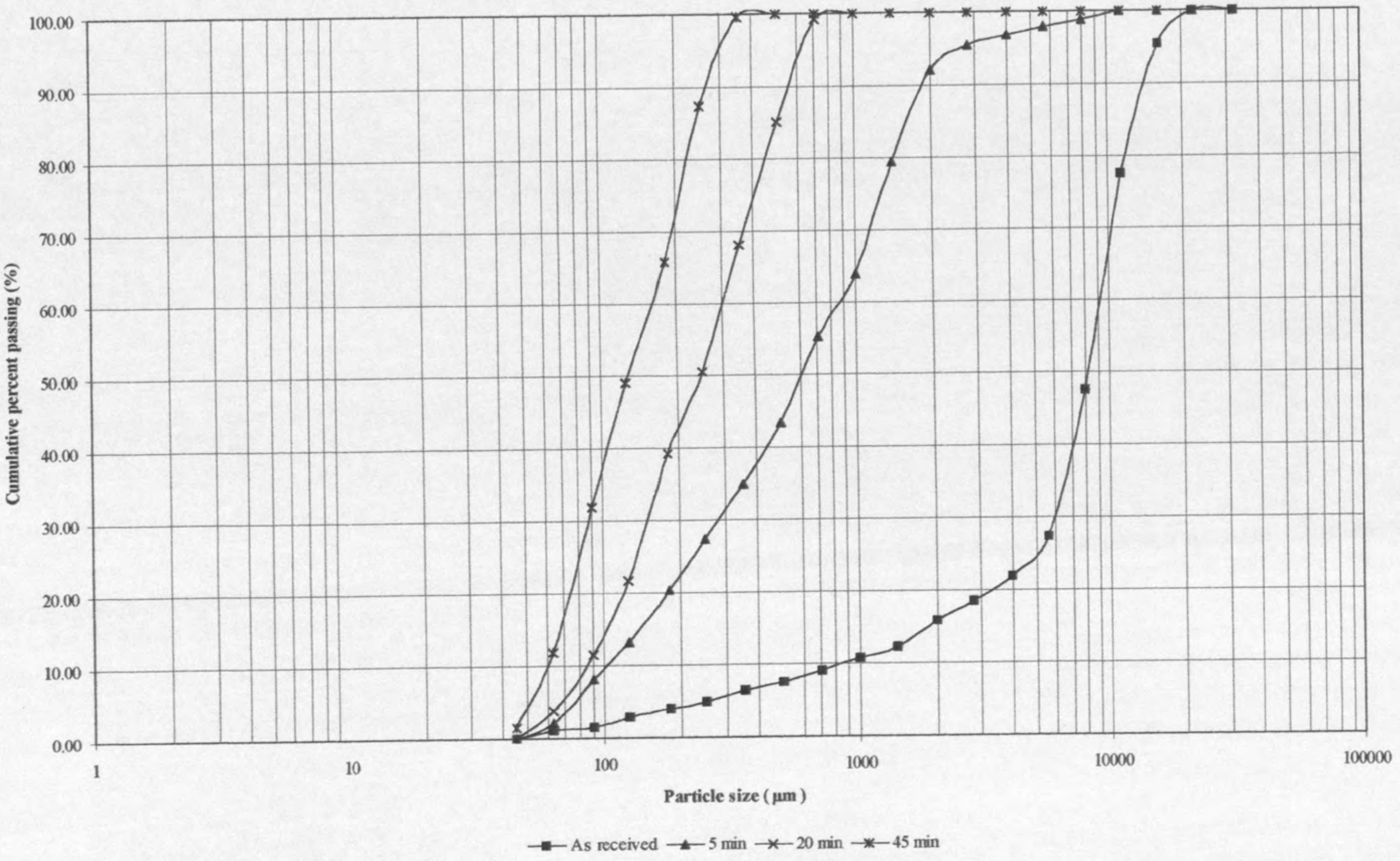
APPENDIX D1

MICROWAVE TREATED (0.65KW) COAL ROD MILL SIZE DISTRIBUTIONS

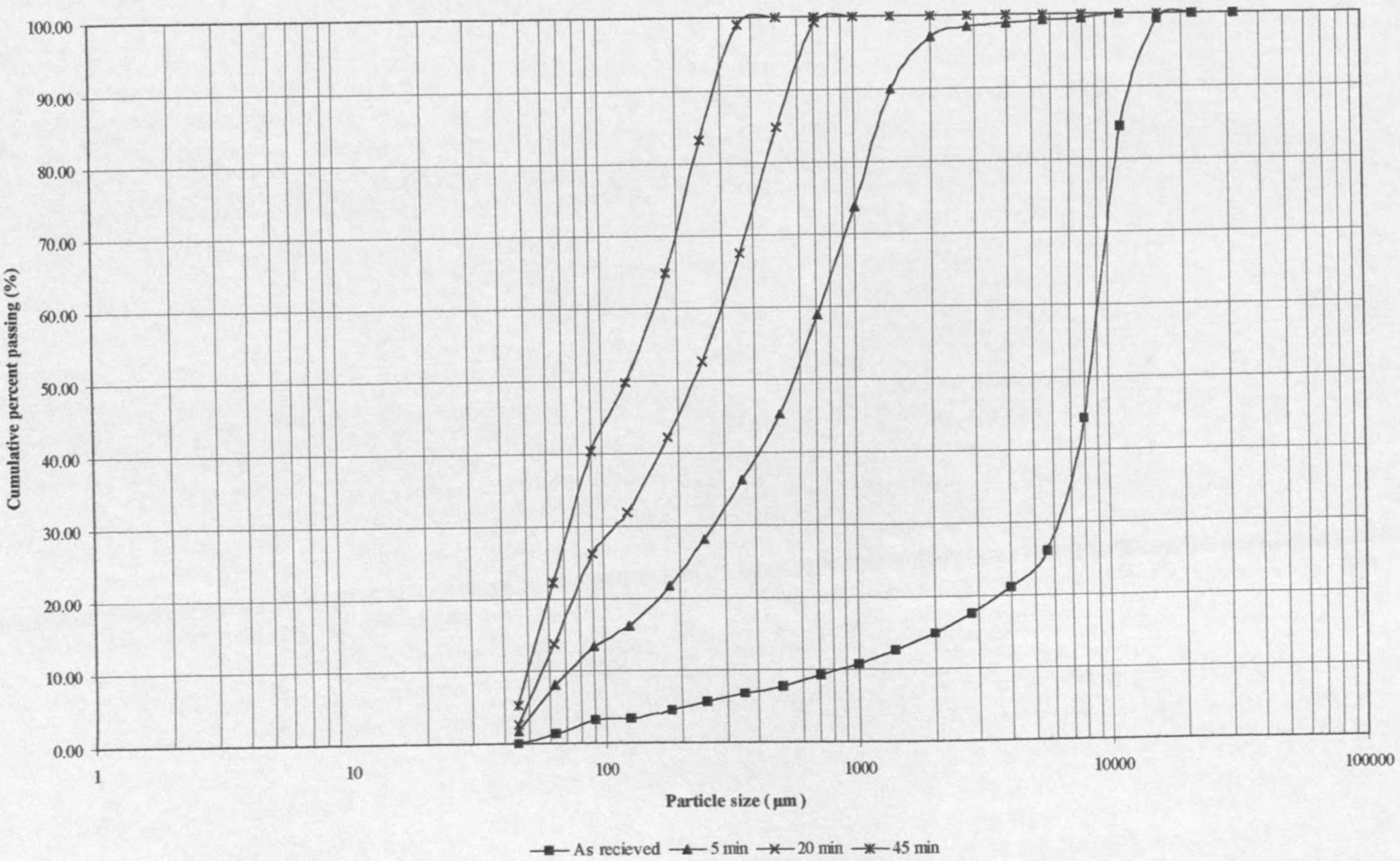
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



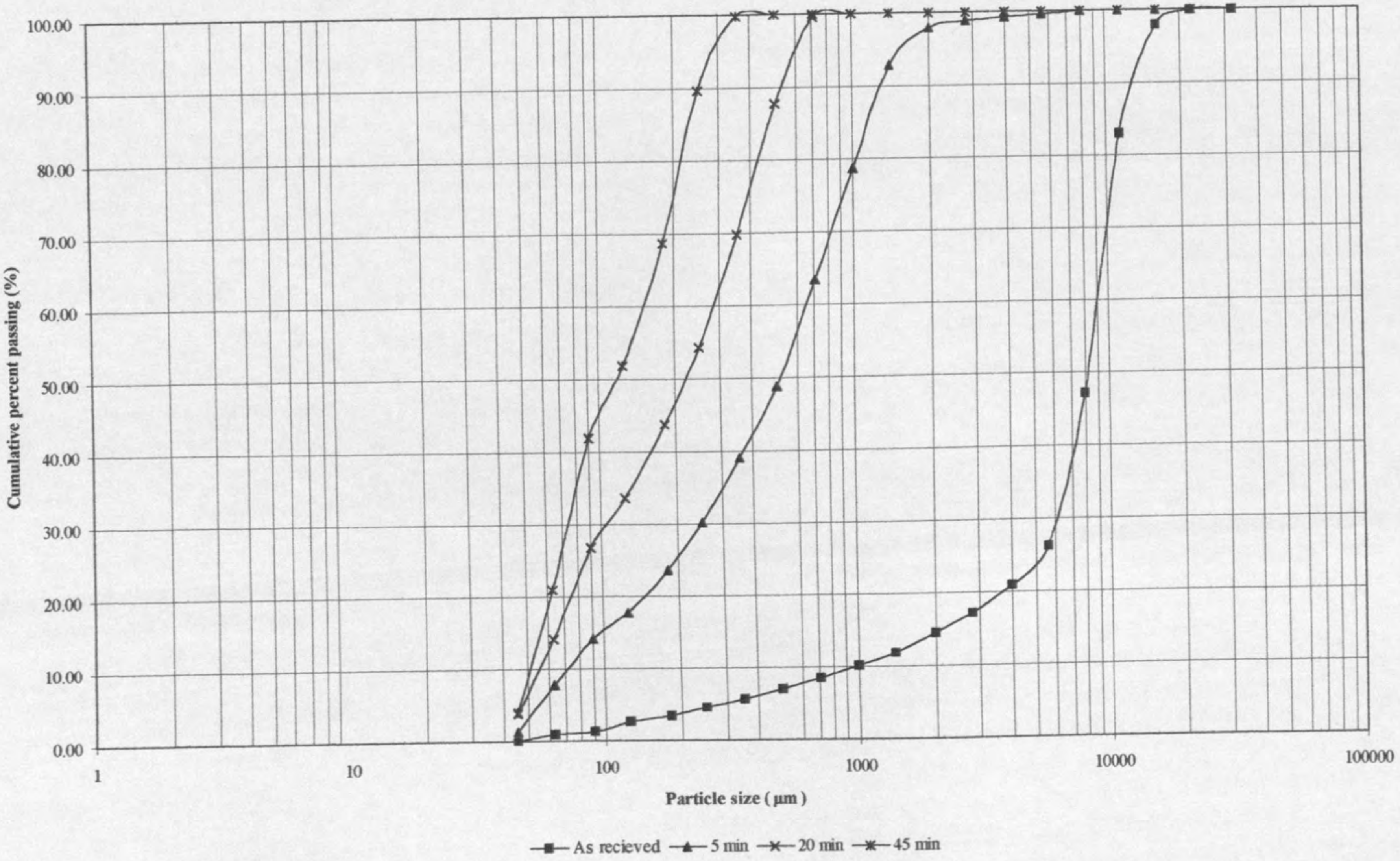
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-1 minute

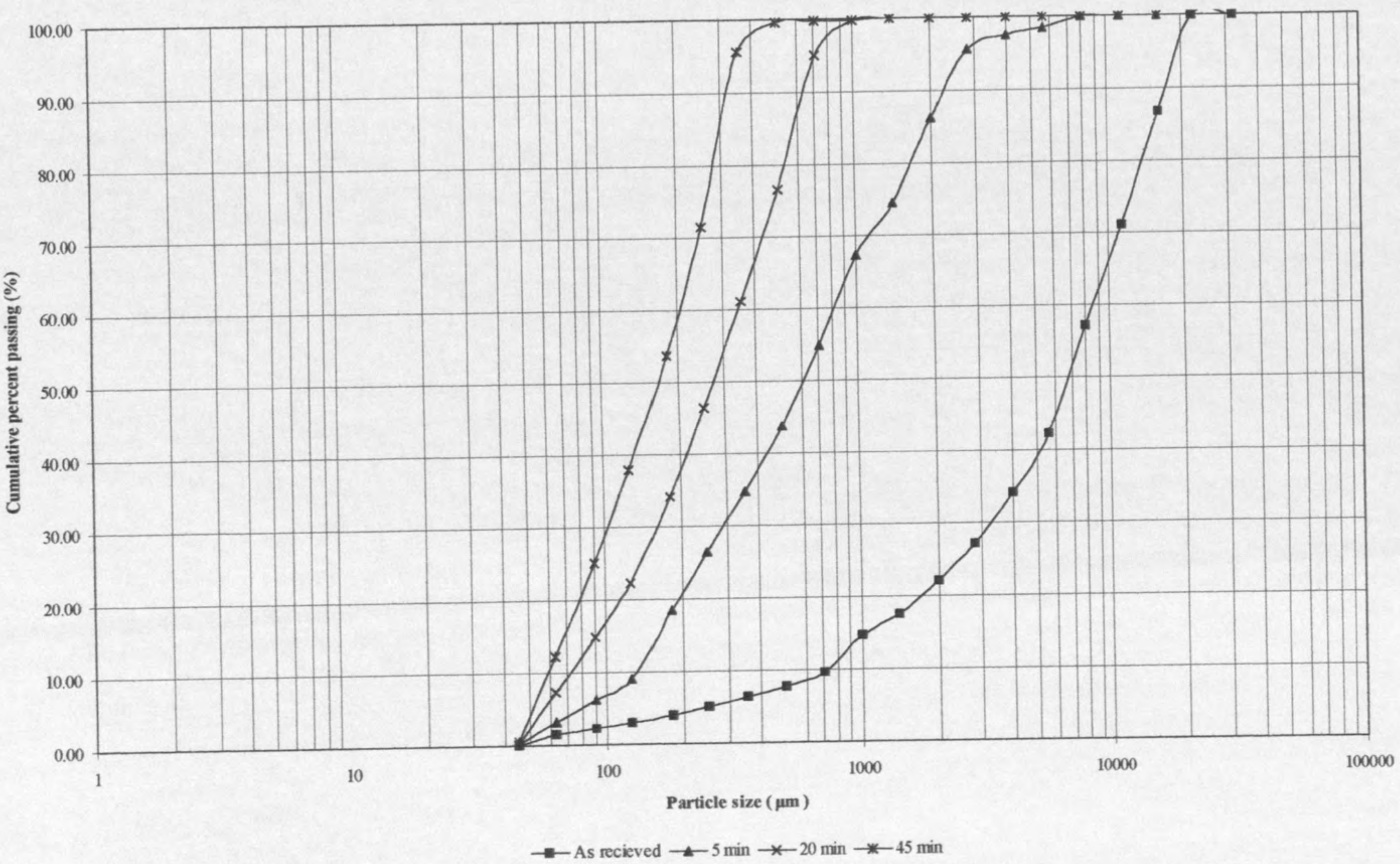


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-3 minute



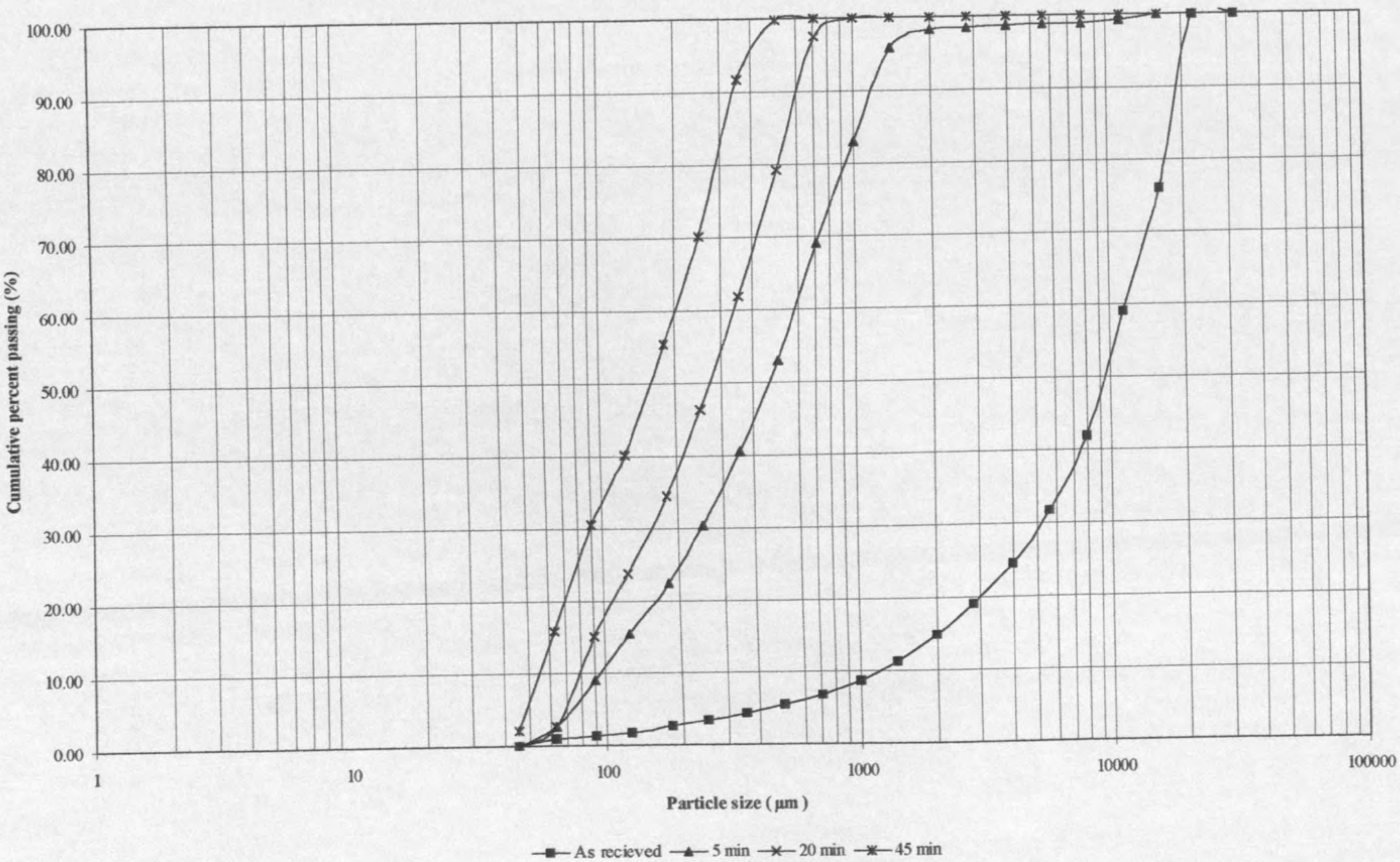
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-8 minute



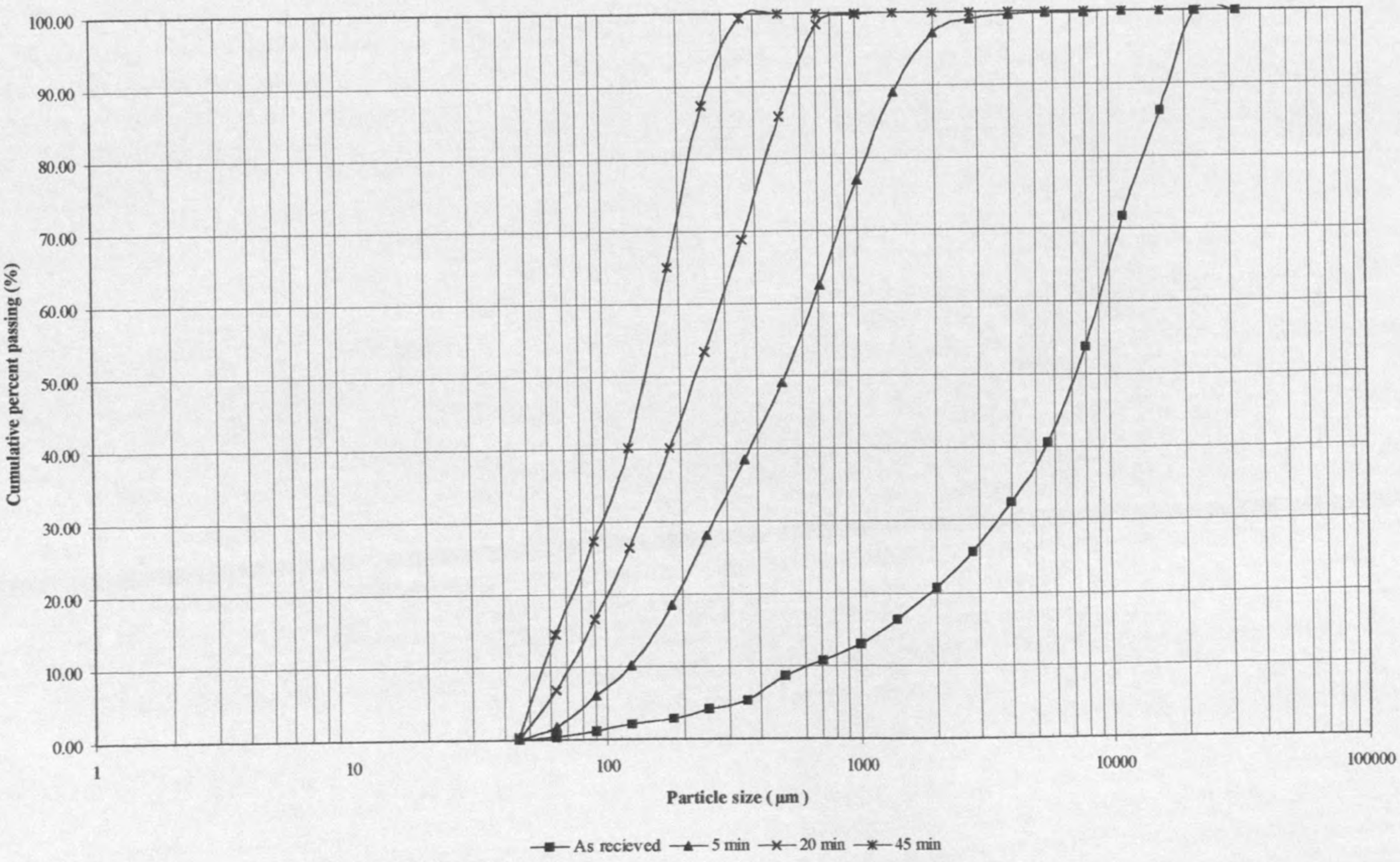


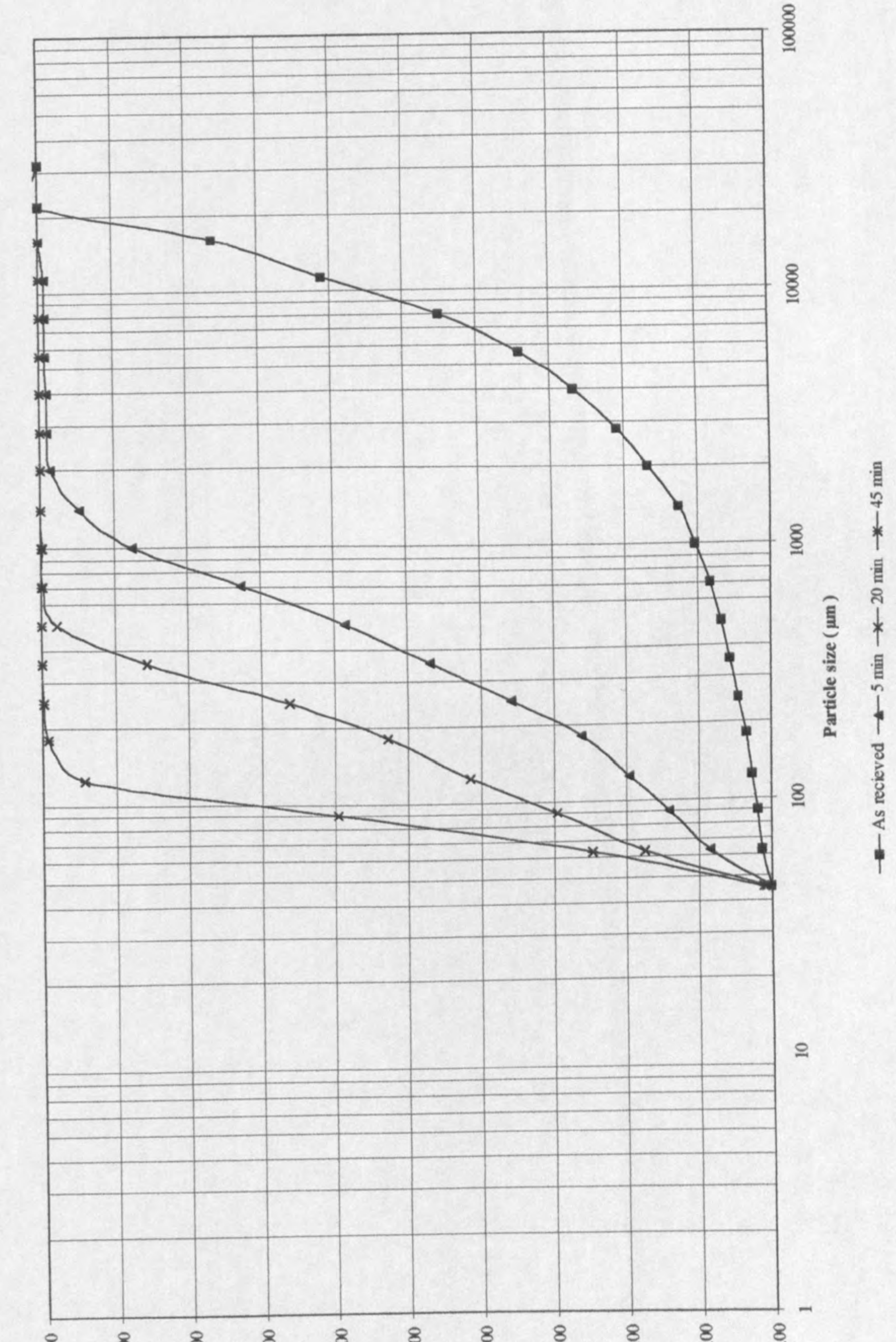
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-1 minute



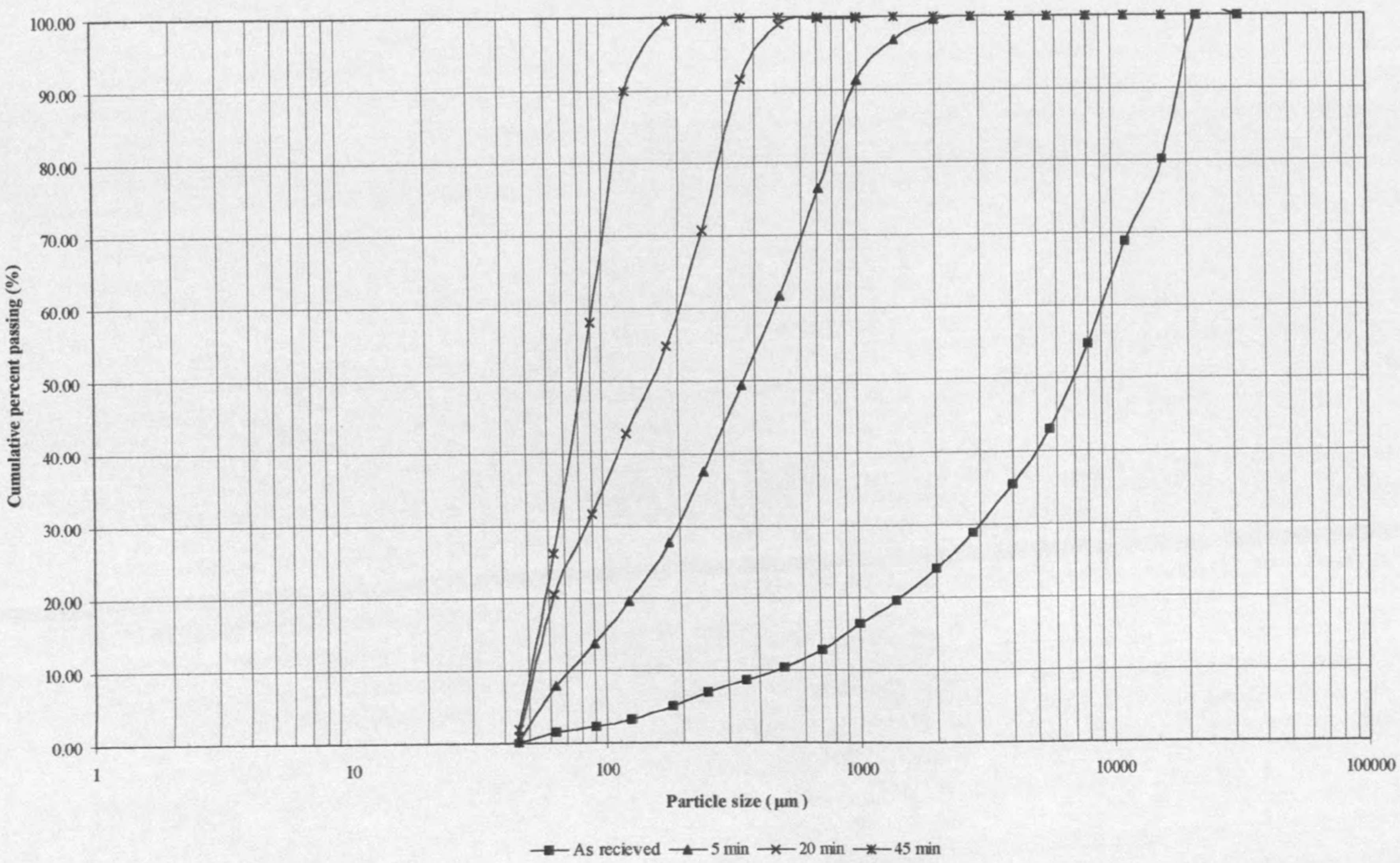
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-3 minute



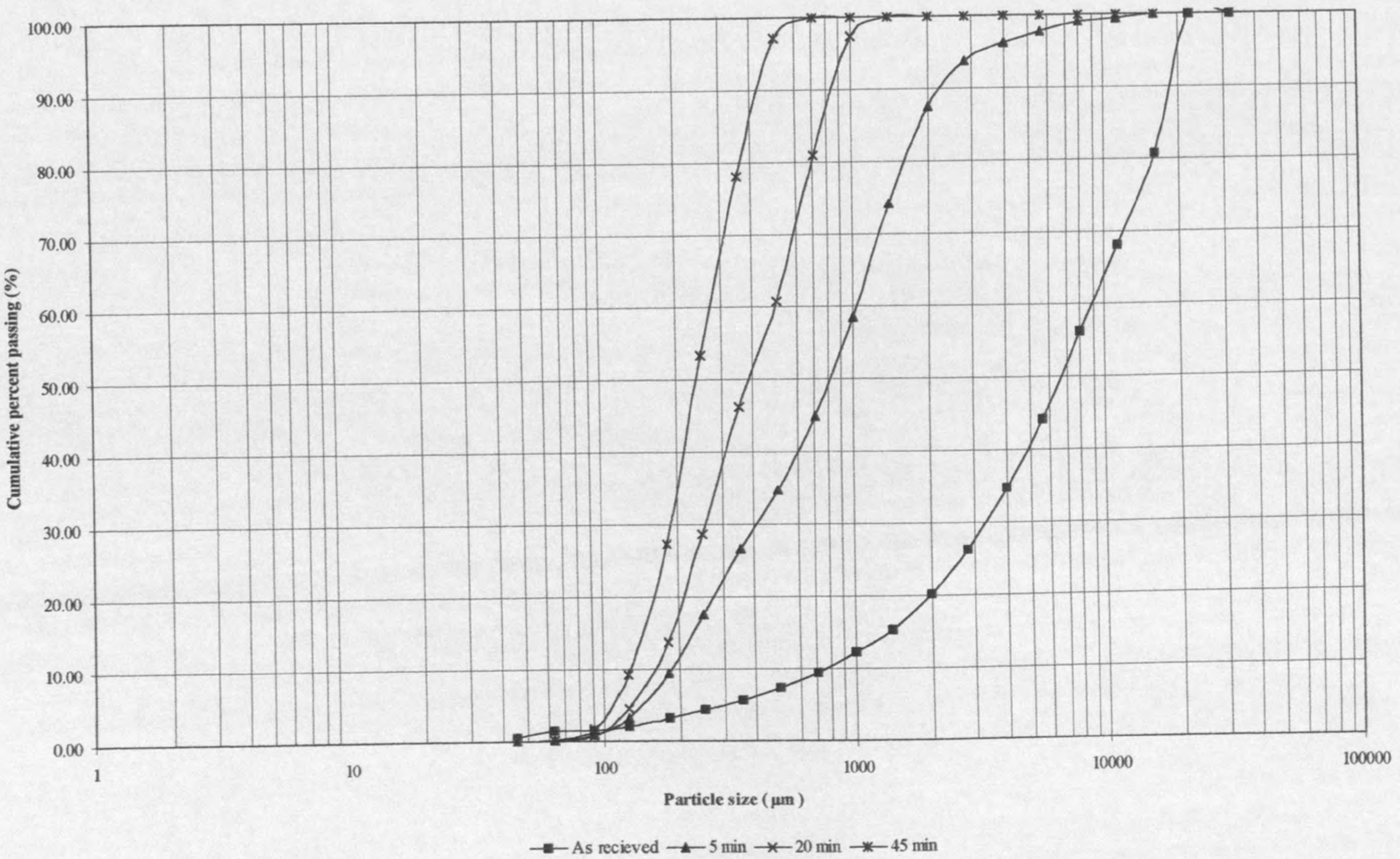


P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-5 minute

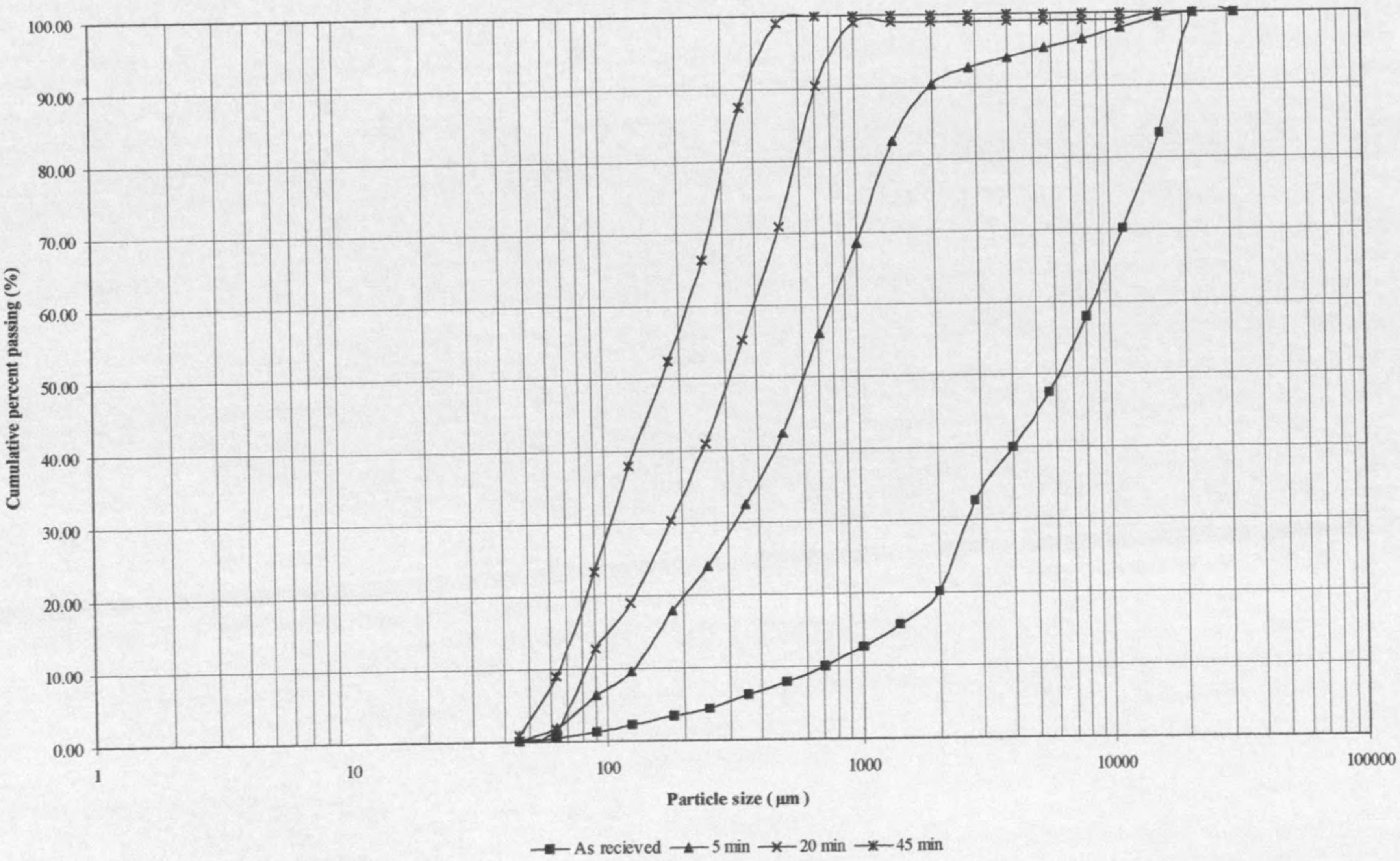
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minute



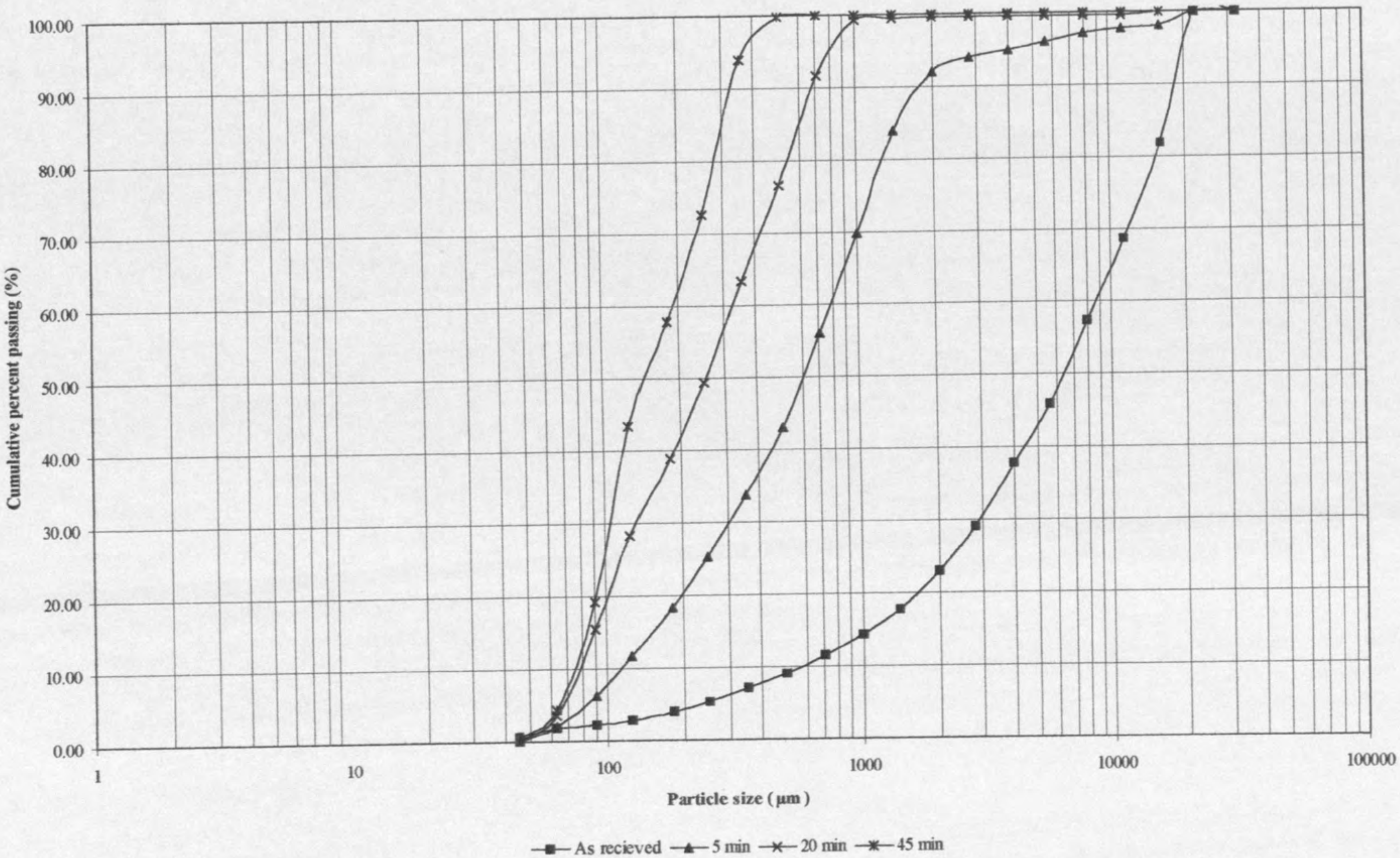
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



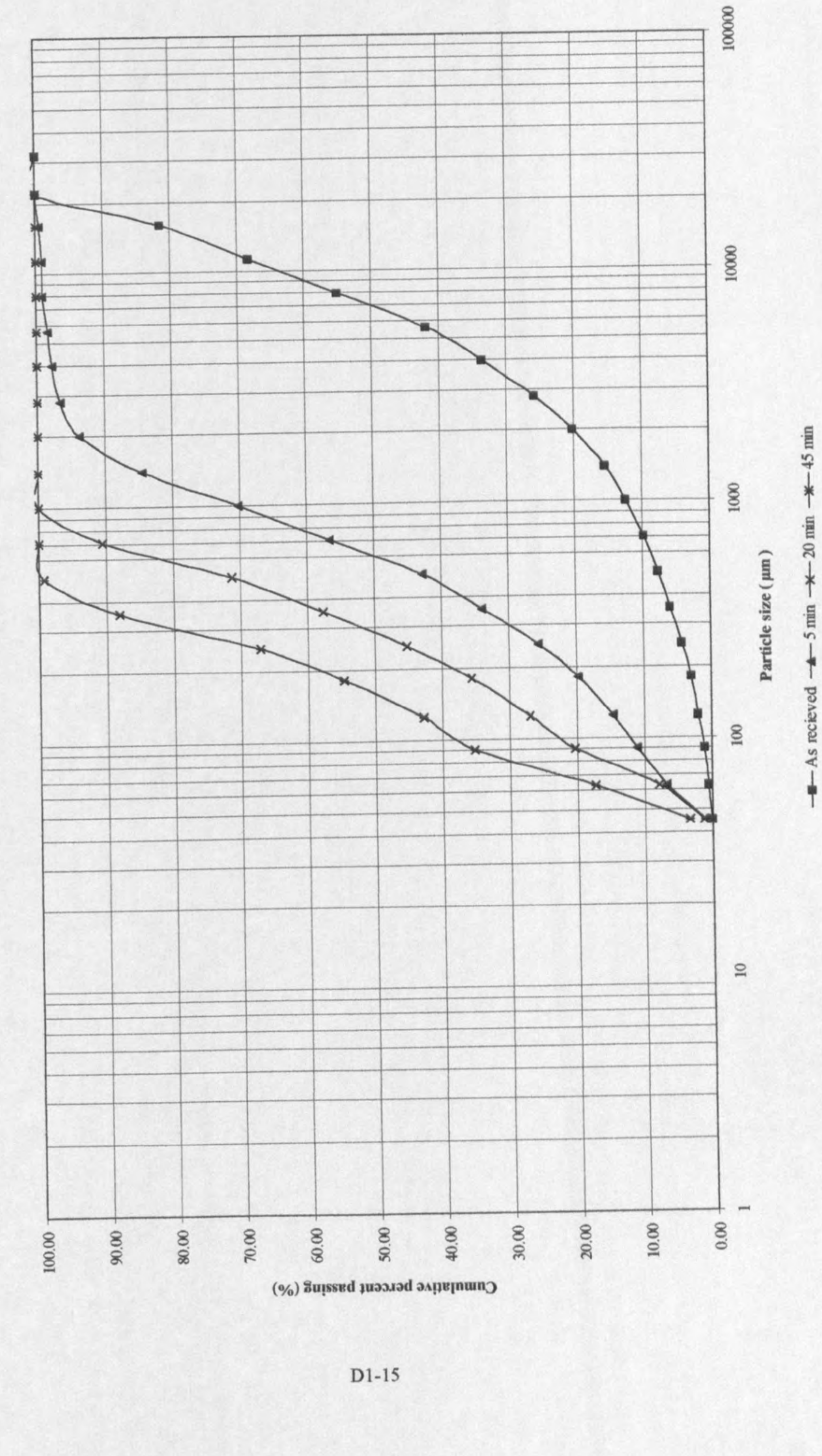
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-1 minute



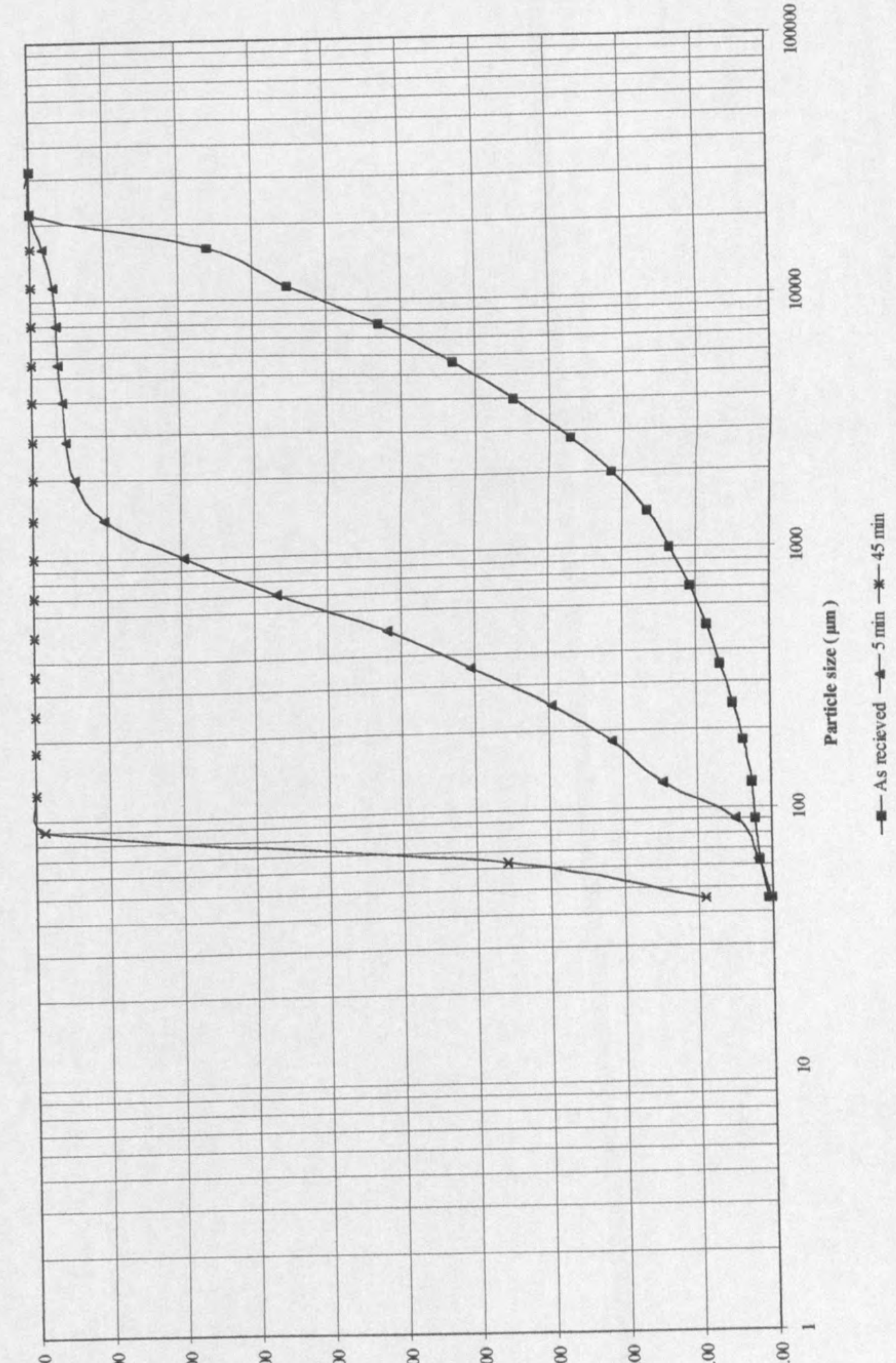
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-3 minute

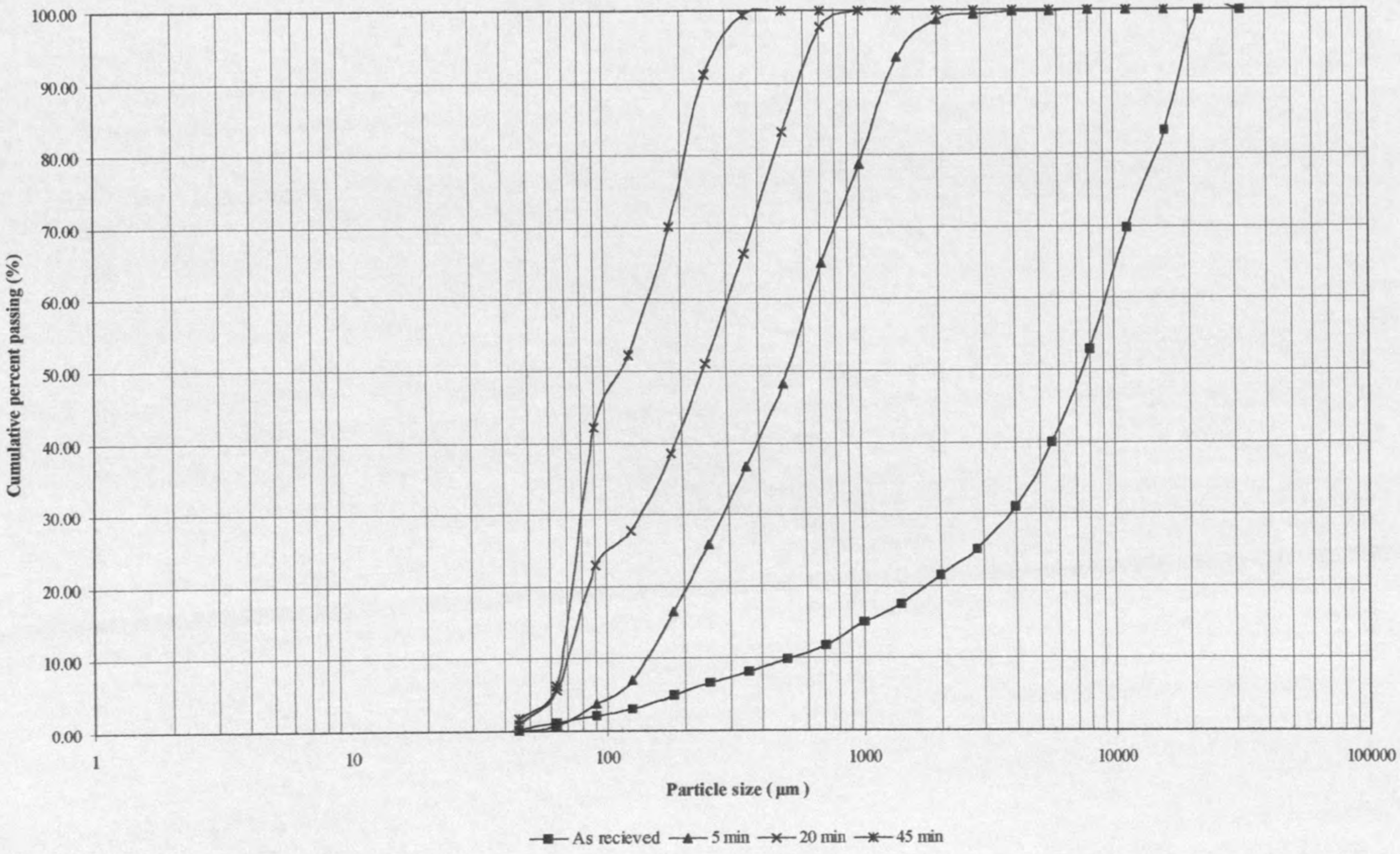


P-2 coal, rod mill, number of rods-7, operating speed-100 rpm, sample load-500 g
Microwave input power-0.65 kW, applied frequency-2.45 GHz, exposure time-5 minute

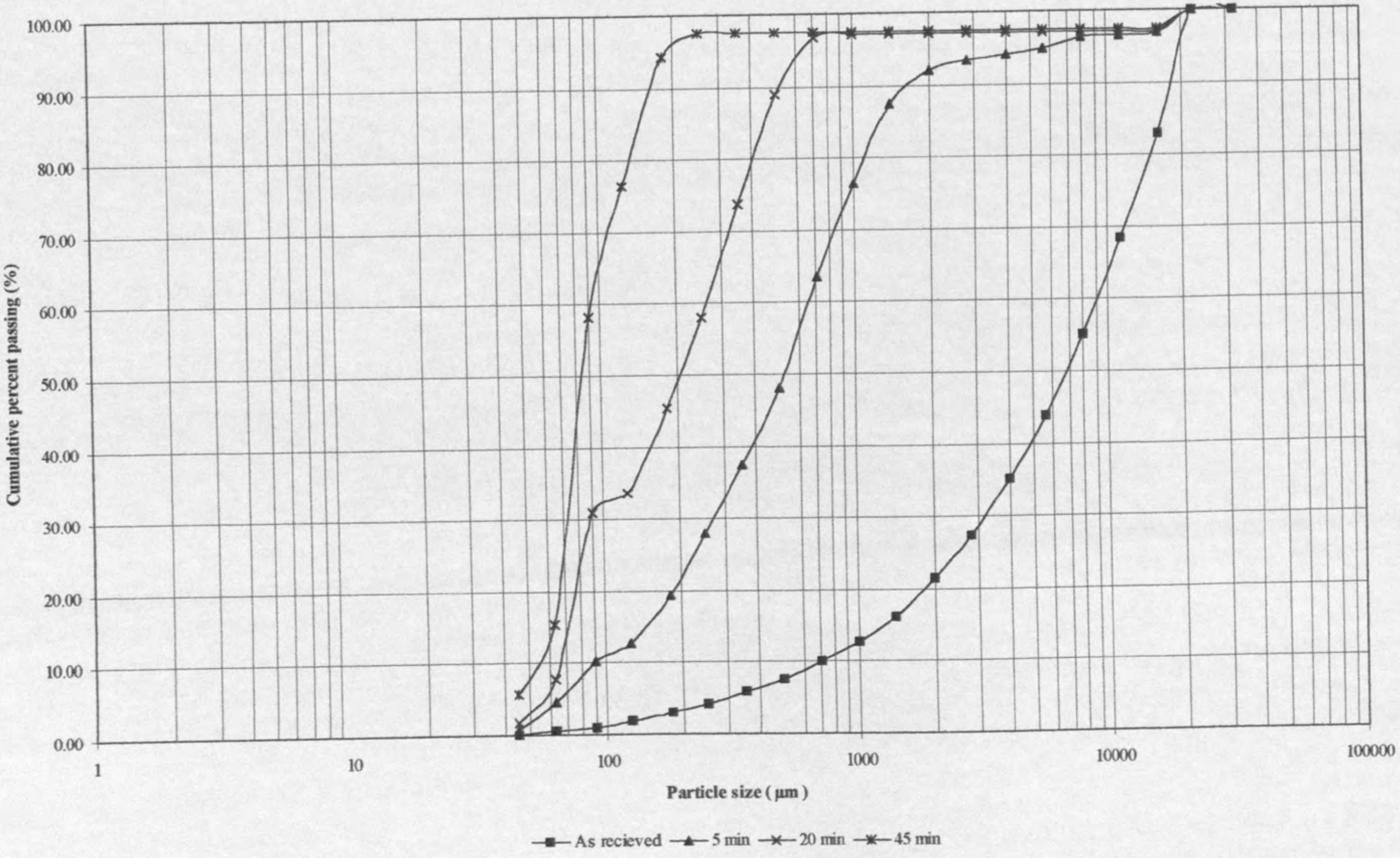


P-2 coal, rod mill, number of rods-7, operating speed-100 rpm, sample load-500 g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minute





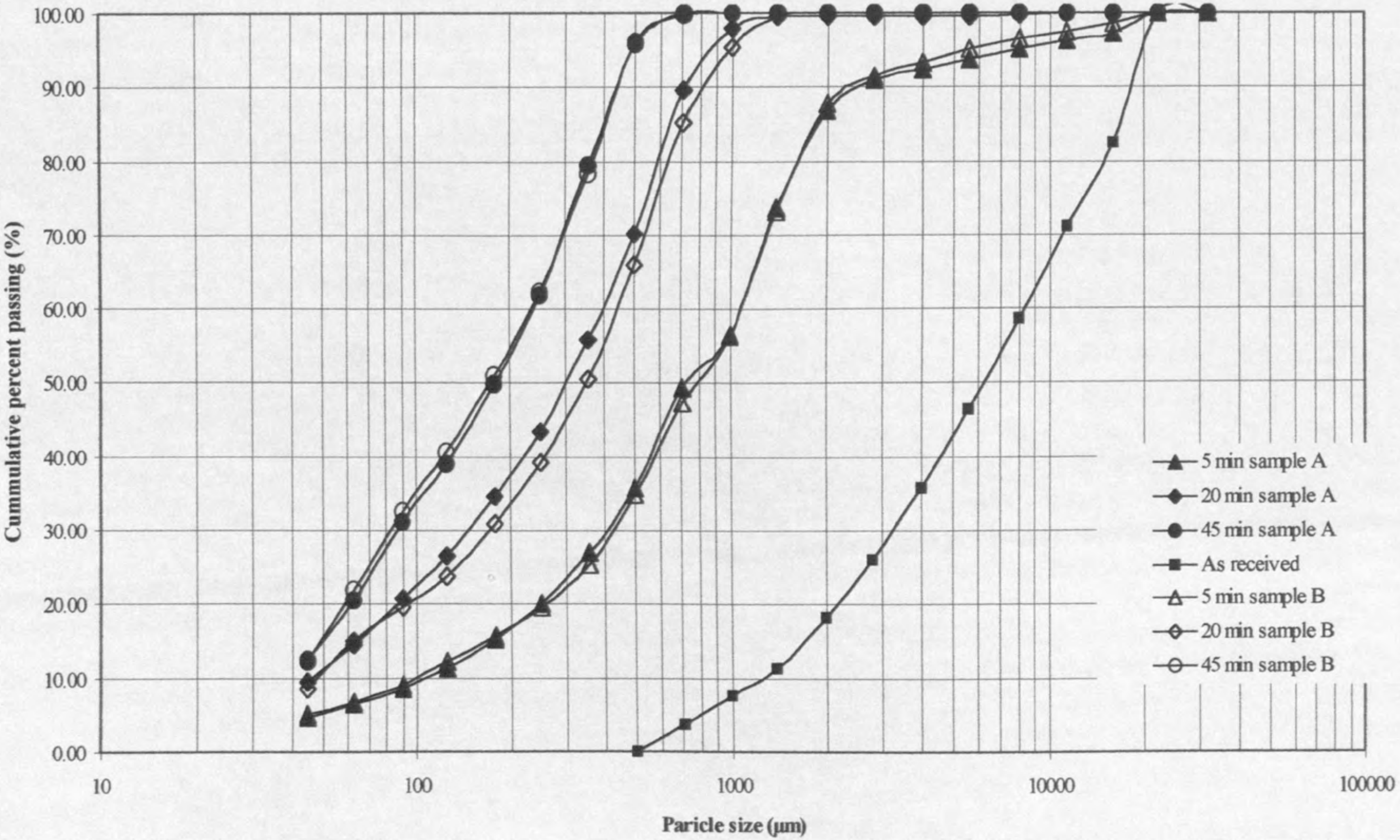
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-250°C, exposed for 1hr


P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-200°C, exposure time-1hr

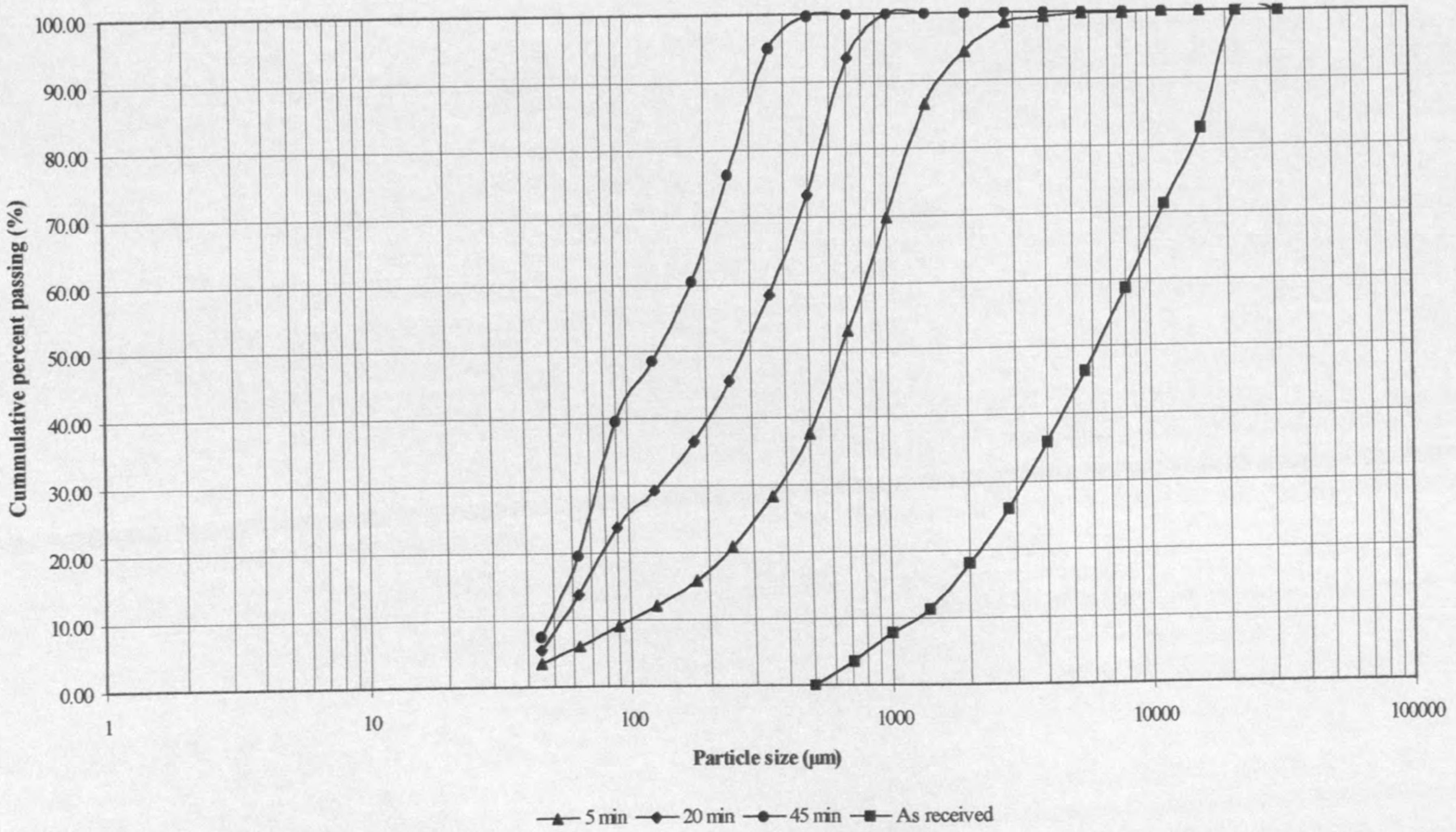
APPENDIX D2

MICROWAVE TREATED (0.65KW) P-2 COAL ROD MILL SIZE DISTRIBUTIONS (UNIFORM FEED CHARACTERISTICS)

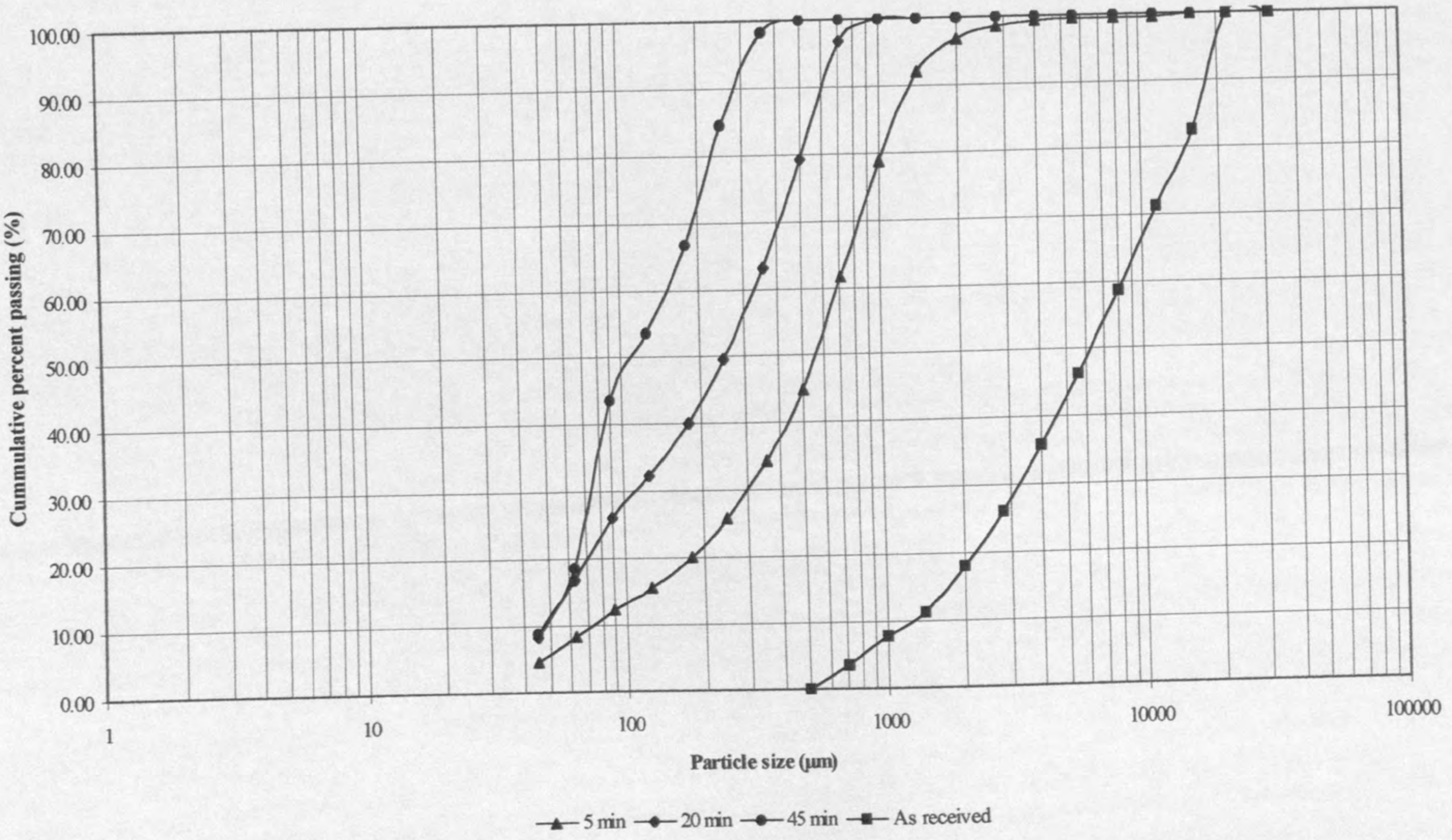
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Size classification of two as-received samples (uniform initial size distribution)



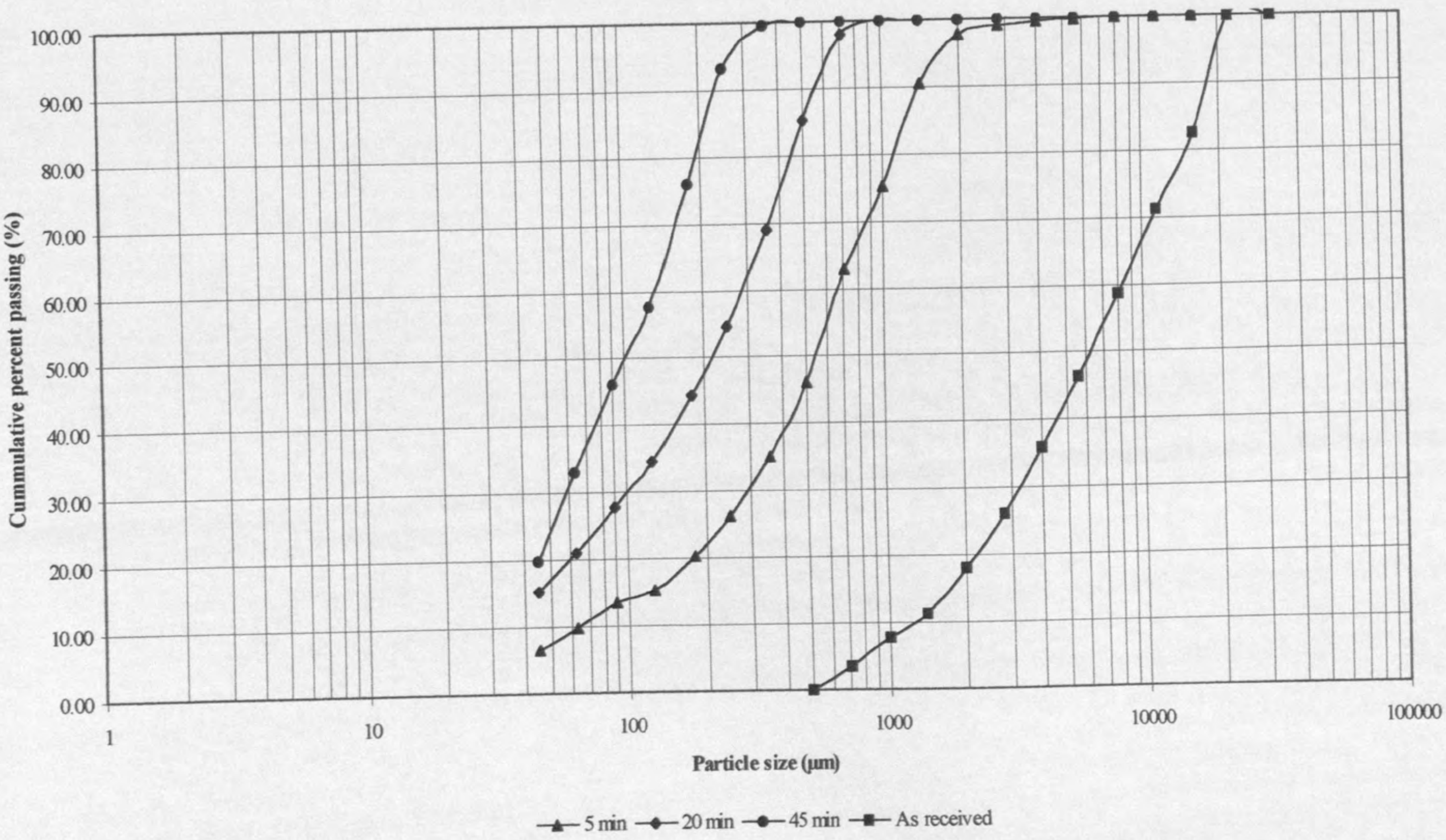
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-3 minutes
(uniform initial size distribution)



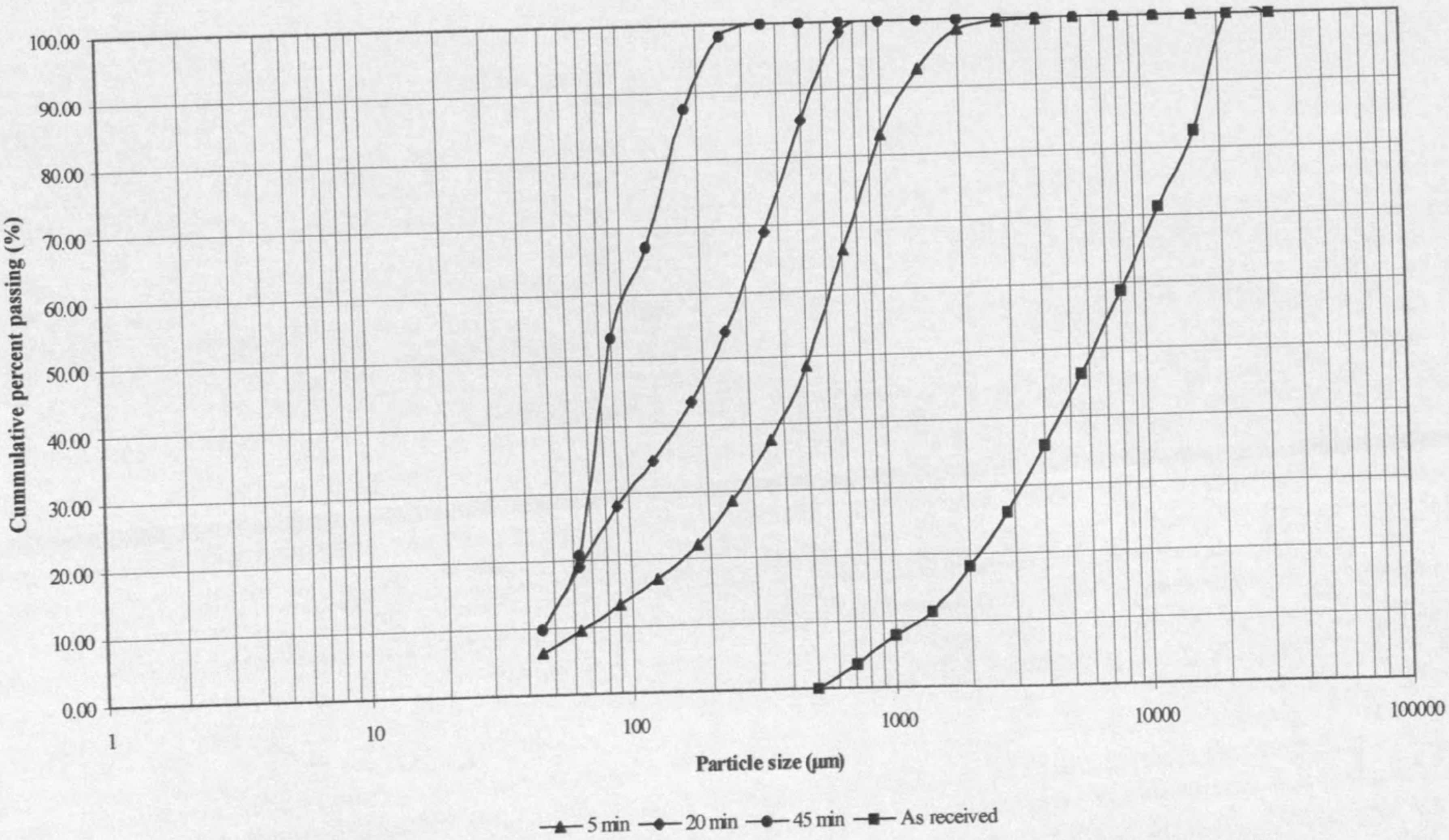
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-5 minutes
(uniform initial size distribution)



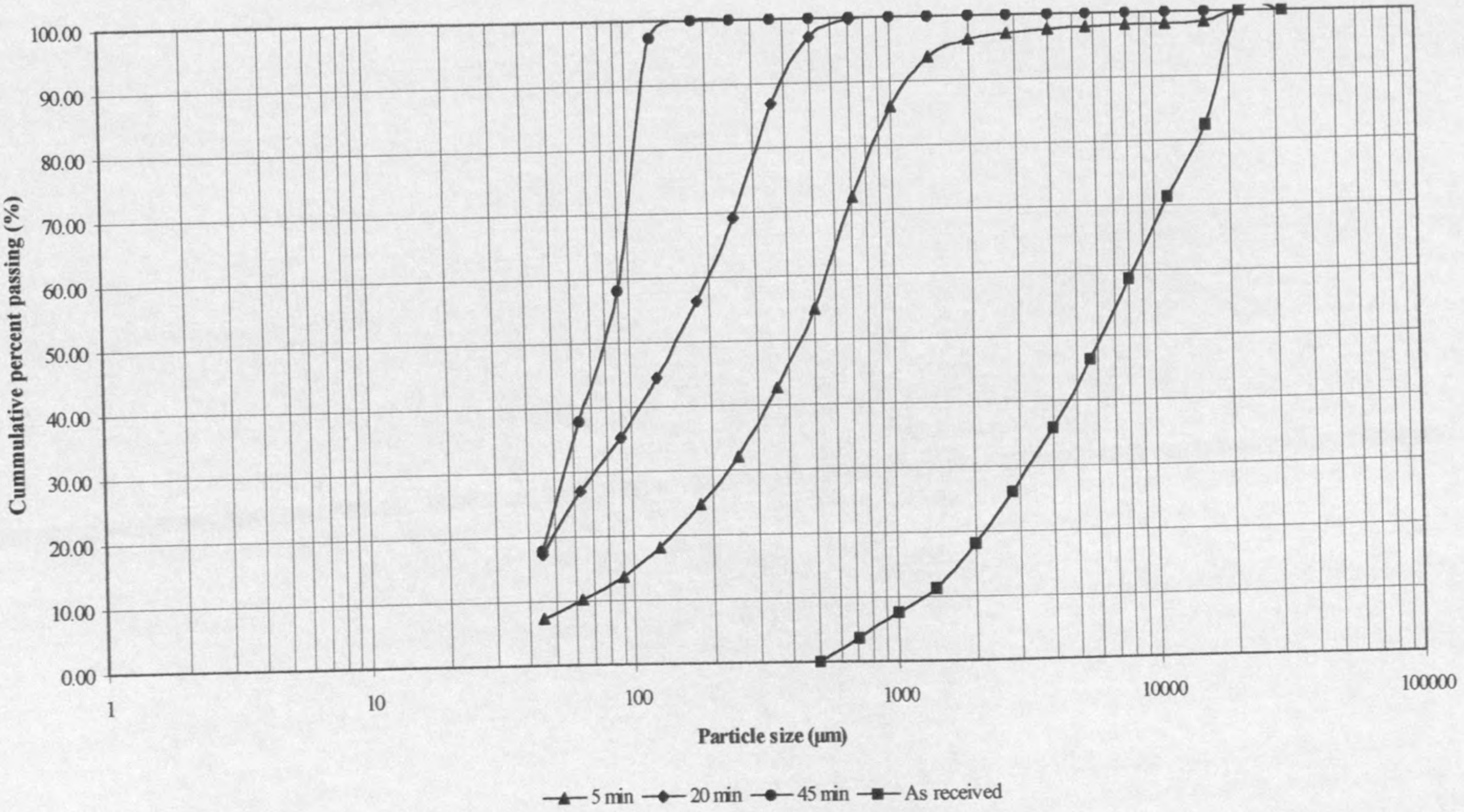
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-8 minutes
(uniform initial size distribution)



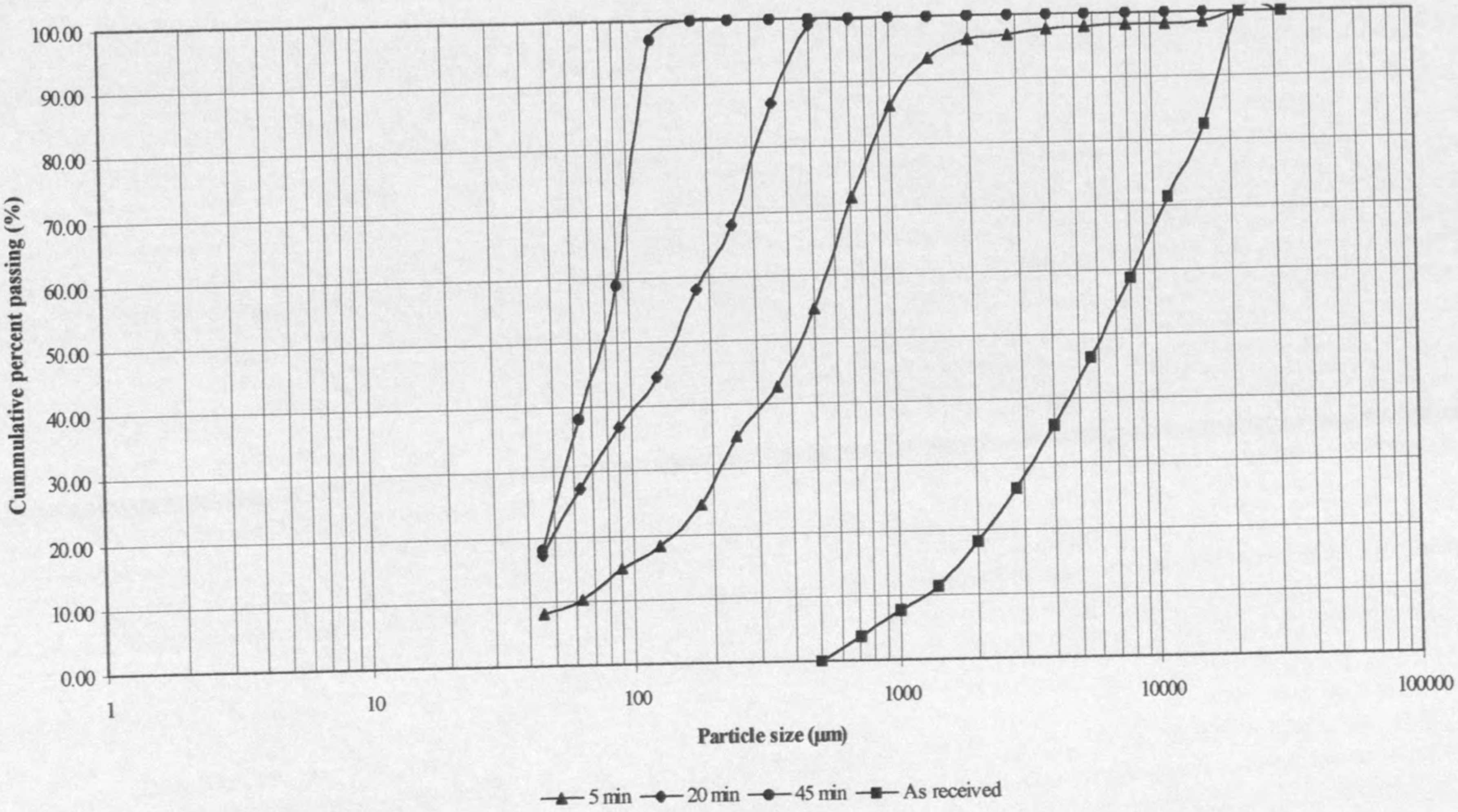
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequancy-2.45GHz, exposure time-10 minutes
(uniform initial size distribution)



P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-12 minutes
(uniform initial size distribution)



P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-15 minutes
(uniform initial size distribution)



APPENDIX D3

PROXIMATE ANALYSIS OF MICROWAVE TREATED COALS

Proximate analysis (as-received basis unless stated in accord with BS1016) of Microwave treated P-5 coal
Microwave input power-0.65kW, applied frequency-2.45GHz

Exposure time (minutes)	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
1	3.7	13.1	34.2	48.9	41.2
3	3.5	10.2	35.0	51.3	40.5
5	3.2	9.3	35.8	51.7	40.9
8	2.6	10.7	34.6	52.1	39.9

Mean value given from 5 trials, Standard deviations given in brackets

Proximate analysis (as-received basis unless stated in accord with BS1016) of Microwave treated P-3 coal
Microwave input power-0.65kW, applied frequency-2.45GHz

Exposure time (minutes)	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
1	4.1	15.1	34.6	46.2	43.3
3	3.0	16.0	34.2	46.8	42.1
5	3.0	16.1	33.0	47.9	40.8
8	2.3	16.1	28.5	53.1	34.9

Mean value given from 5 trials, Standard deviations given in brackets

Proximate analysis (as-received basis unless stated in accord with BS1016) of Microwave treated P-2 coal
Microwave input power-0.65kW, applied frequency-2.45GHz

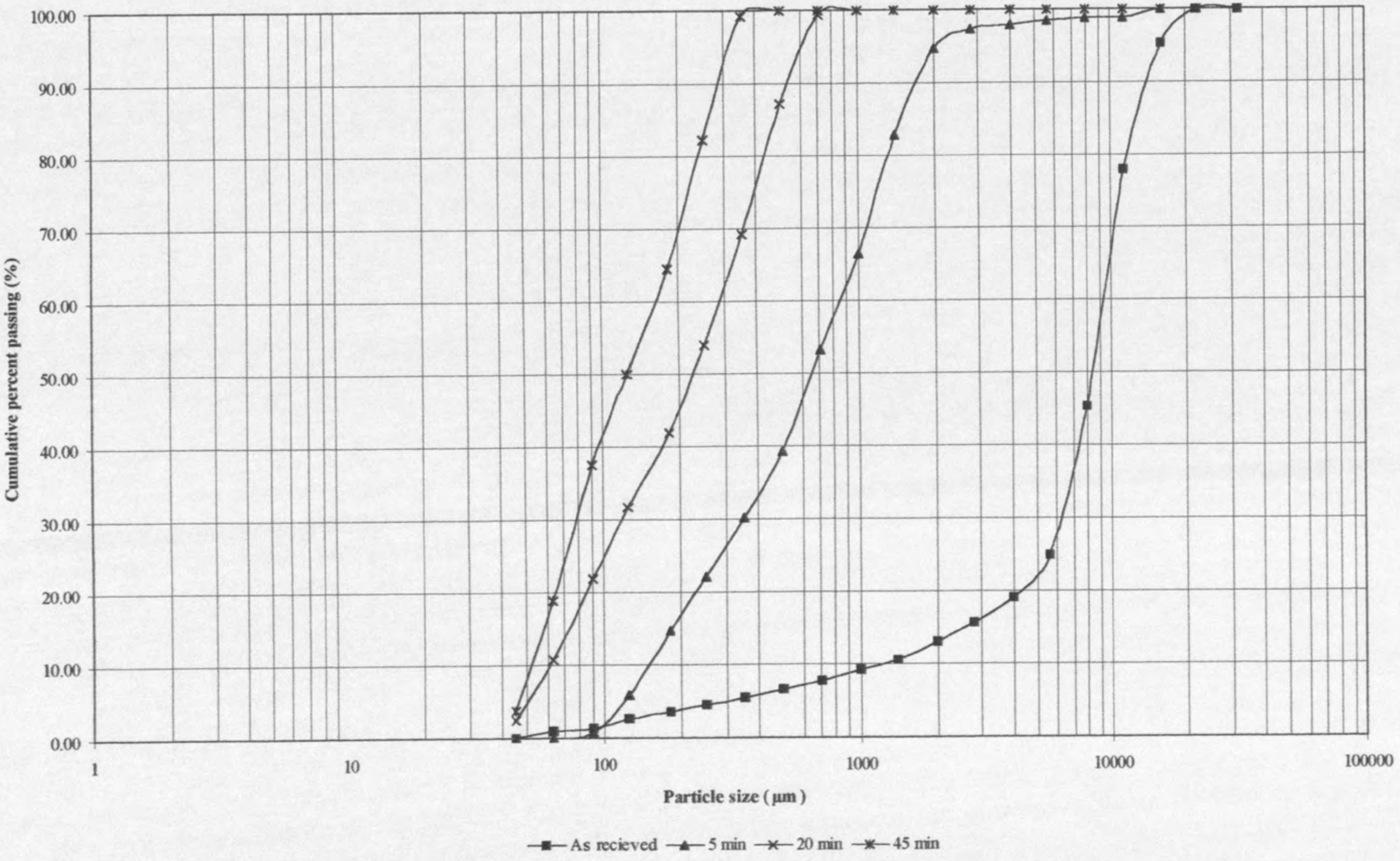
Exposure time (minutes)	Moisture %	Ash %	Volatile matter %	Fixed Carbon %	Volatile matter $_{dmmf}$ %
1	4.4	14.0	31.6	50.0	38.7
3	4.1	11.4	30.9	53.6	36.6
5	3.9	10.9	31.0	54.2	36.4
8	2.3	15.2	30.8	51.7	37.3

Mean value given from 5 trials, Standard deviations given in brackets

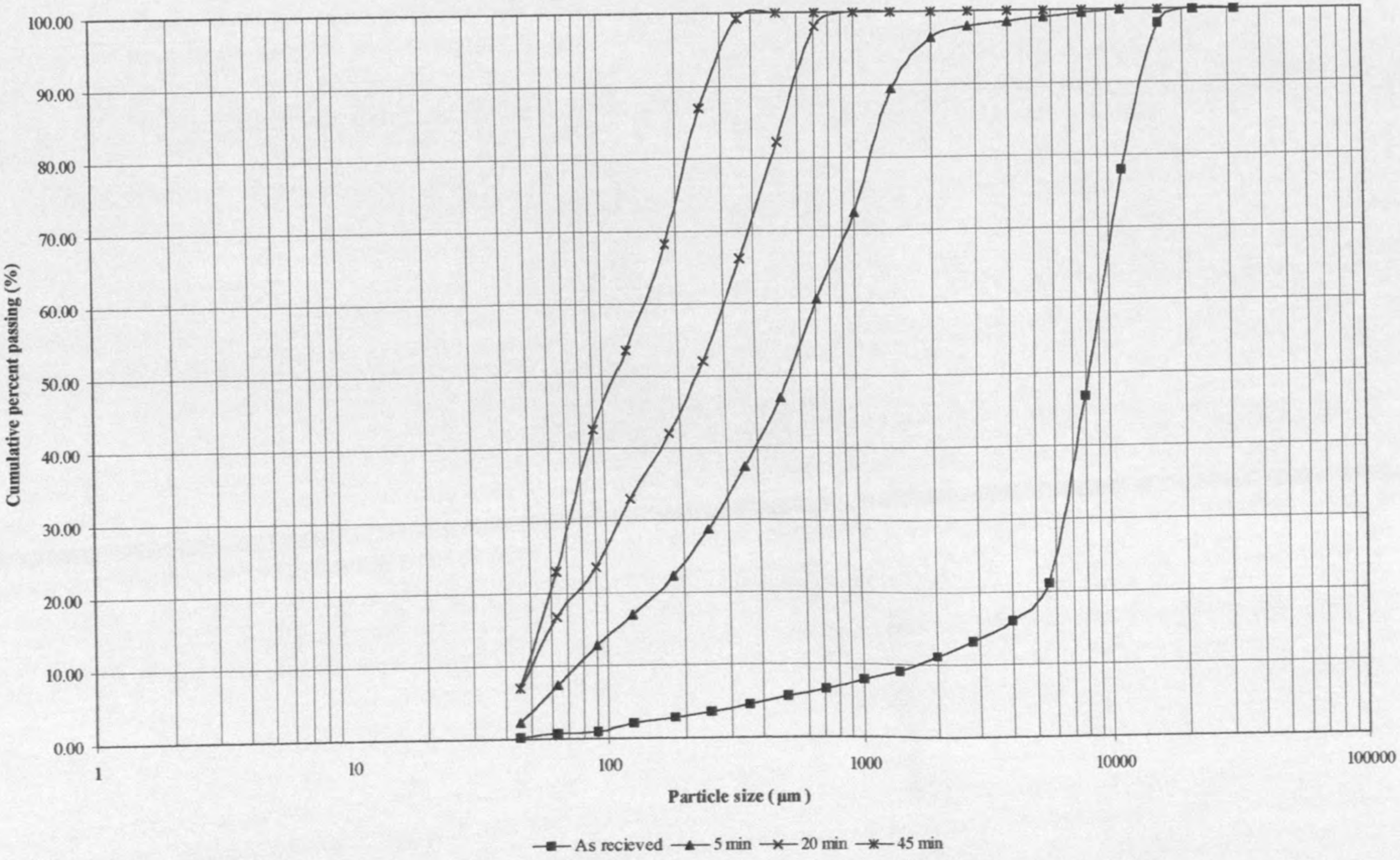
APPENDIX D4

MICROWAVE TREATED (1.3KW) COAL ROD MILL SIZE DISTRIBUTIONS

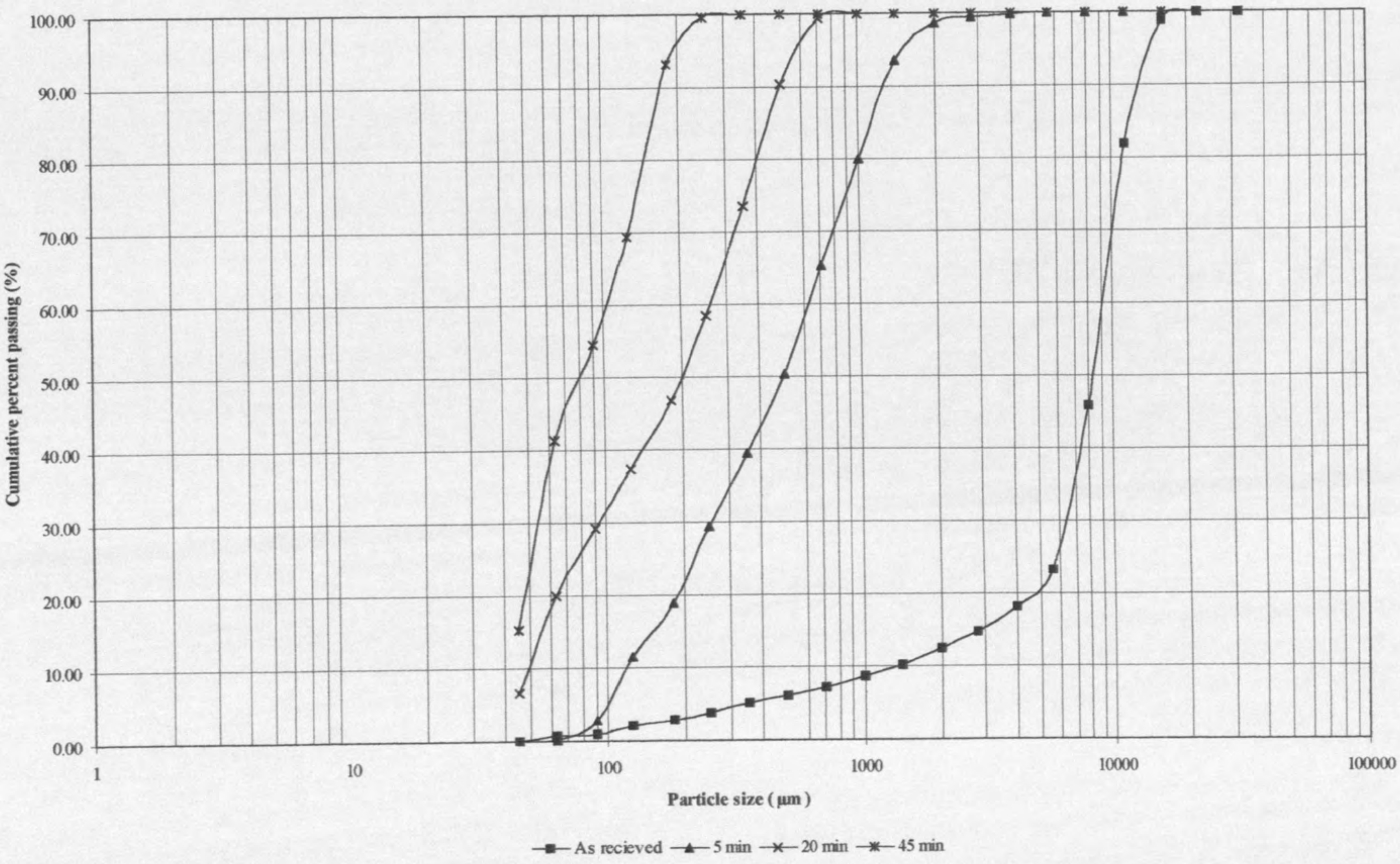
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequancy-2.45GHz, exposure time-1 minute

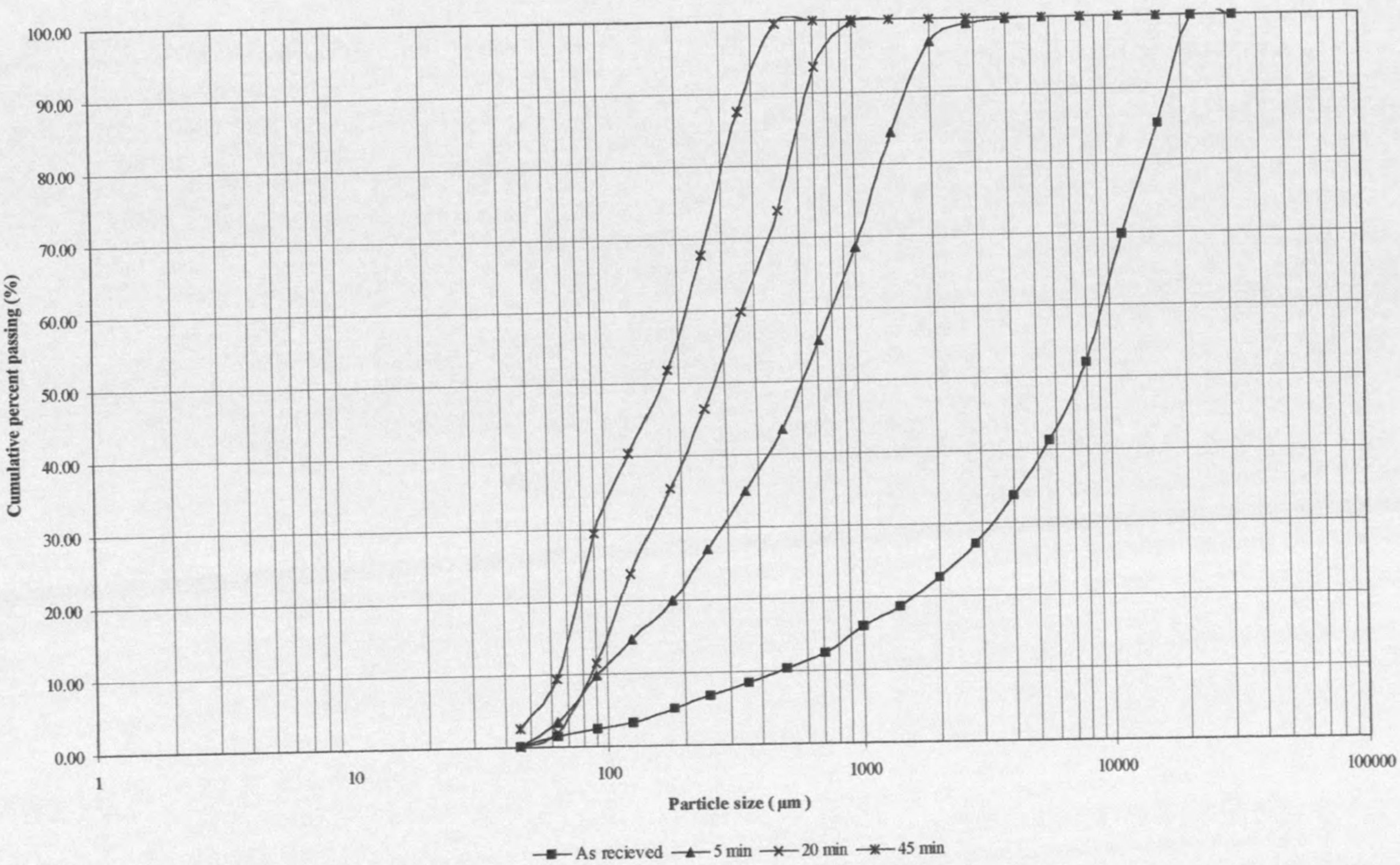


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequancy-2.45GHz, exposure time-2 minute



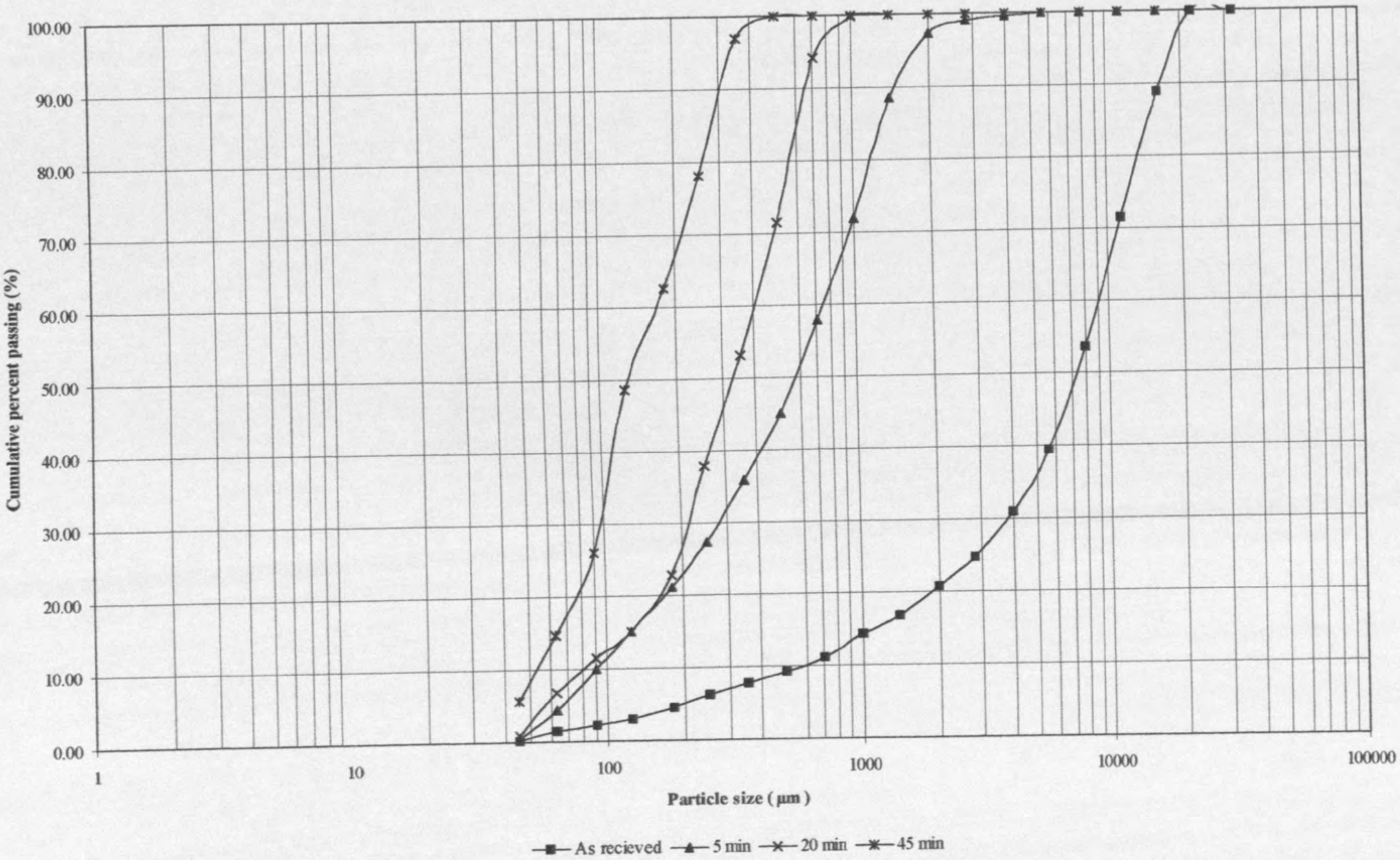
P-5 coal, rod mill, number of rods-7, operating speed-100 rpm, sample load-500g
Microwave input power-1.3kW, applied frequancy-2.45GHz, exposure time-2.5 minute



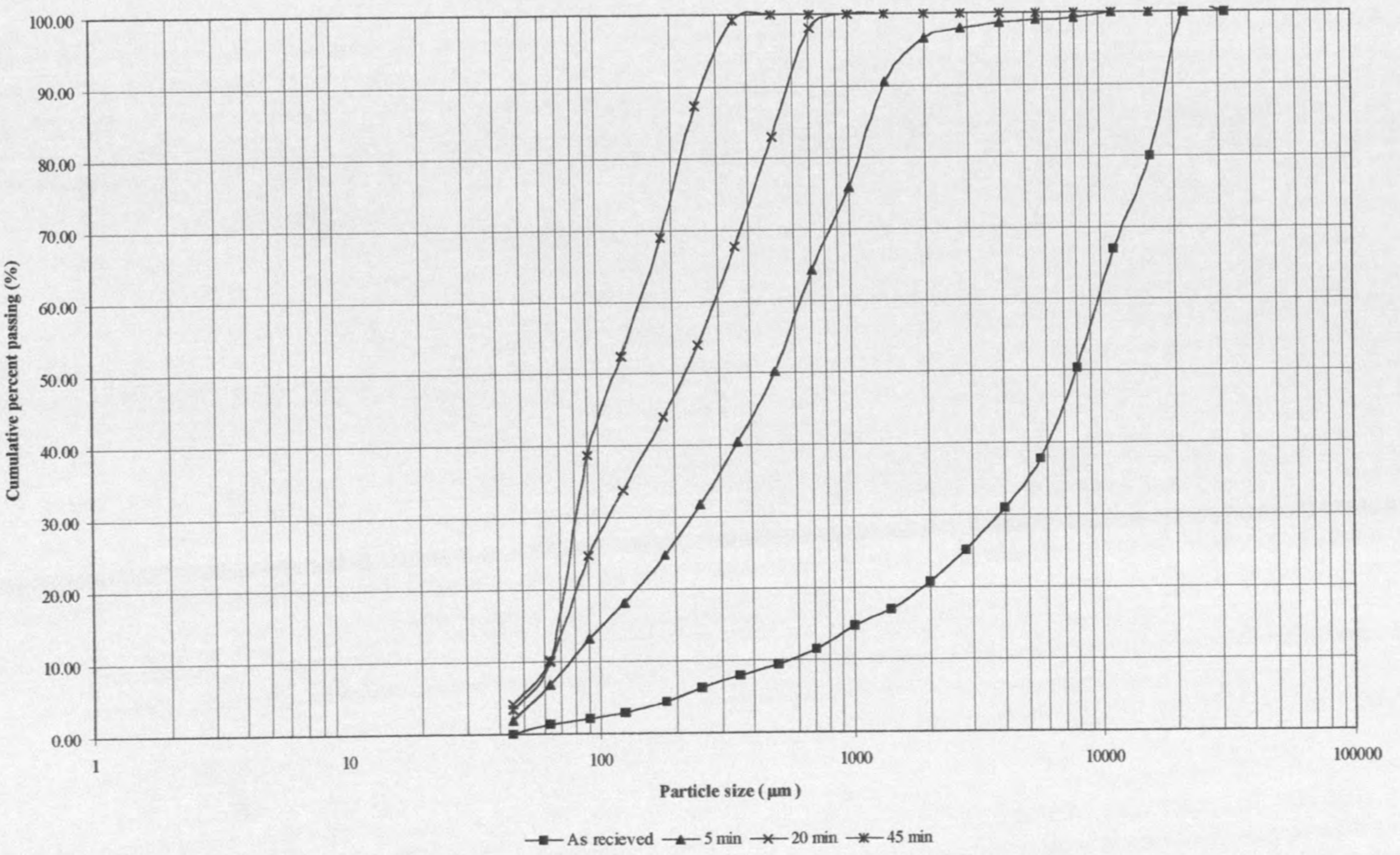


P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-1 minute

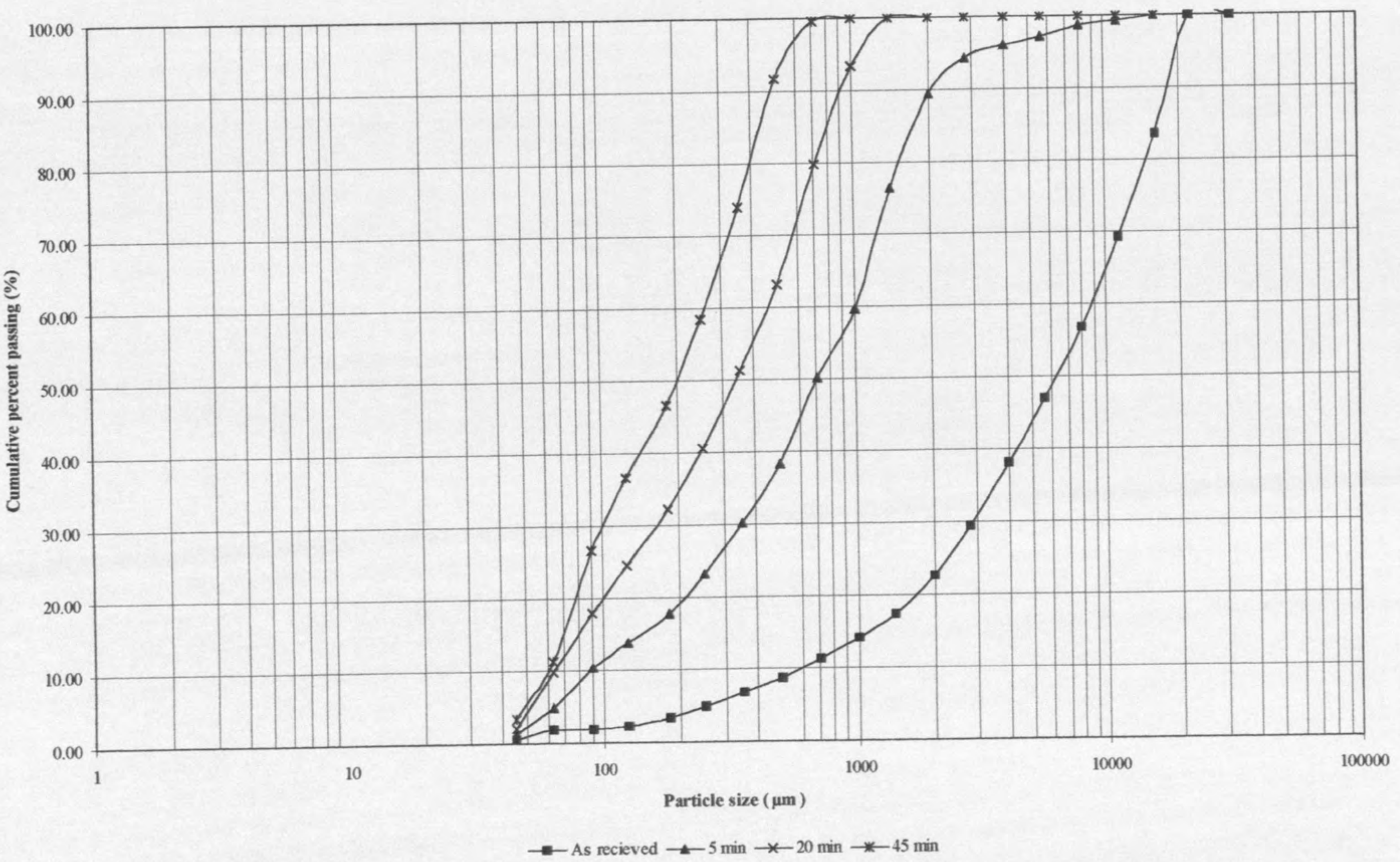
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-2 minute



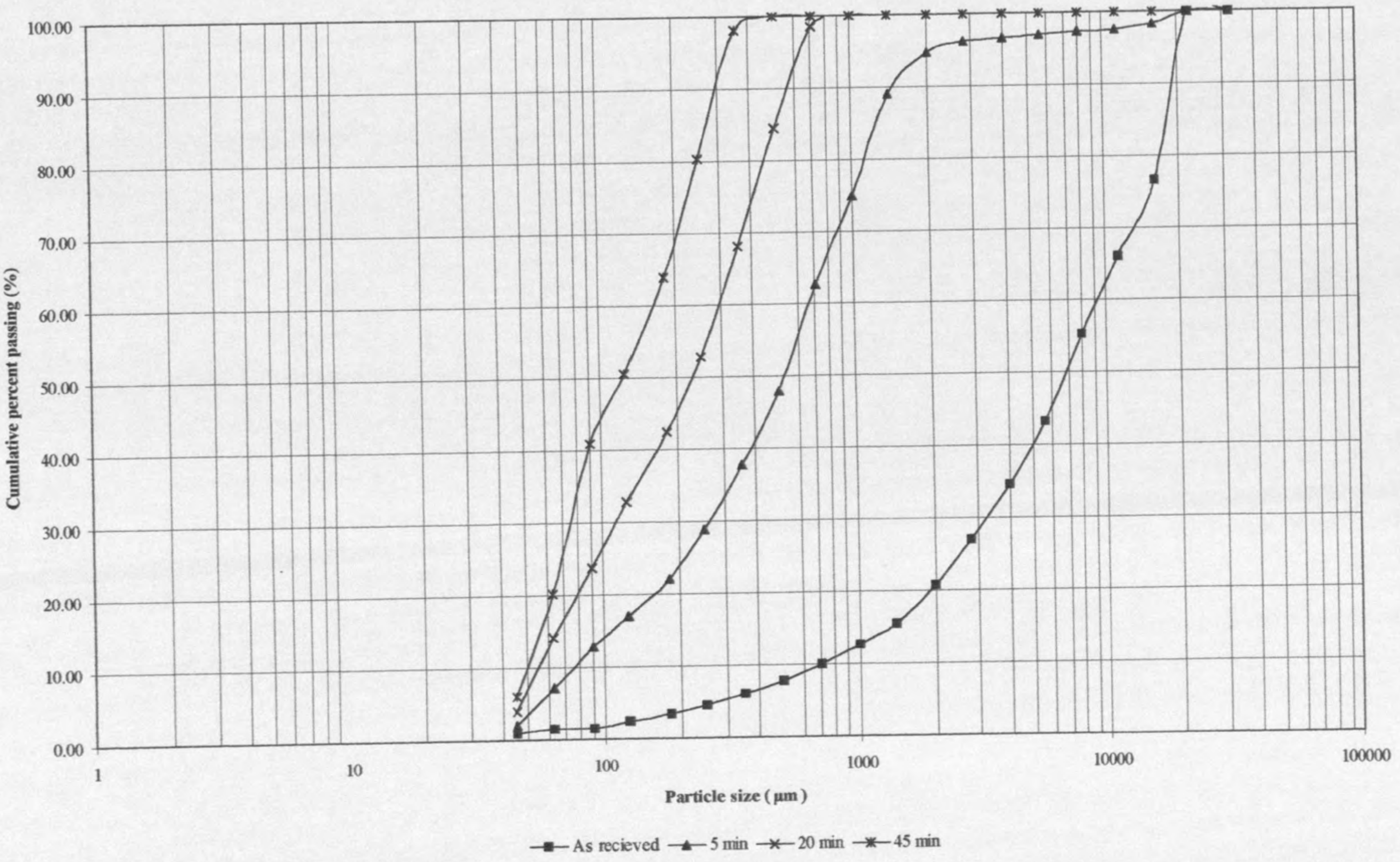
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-3 minute



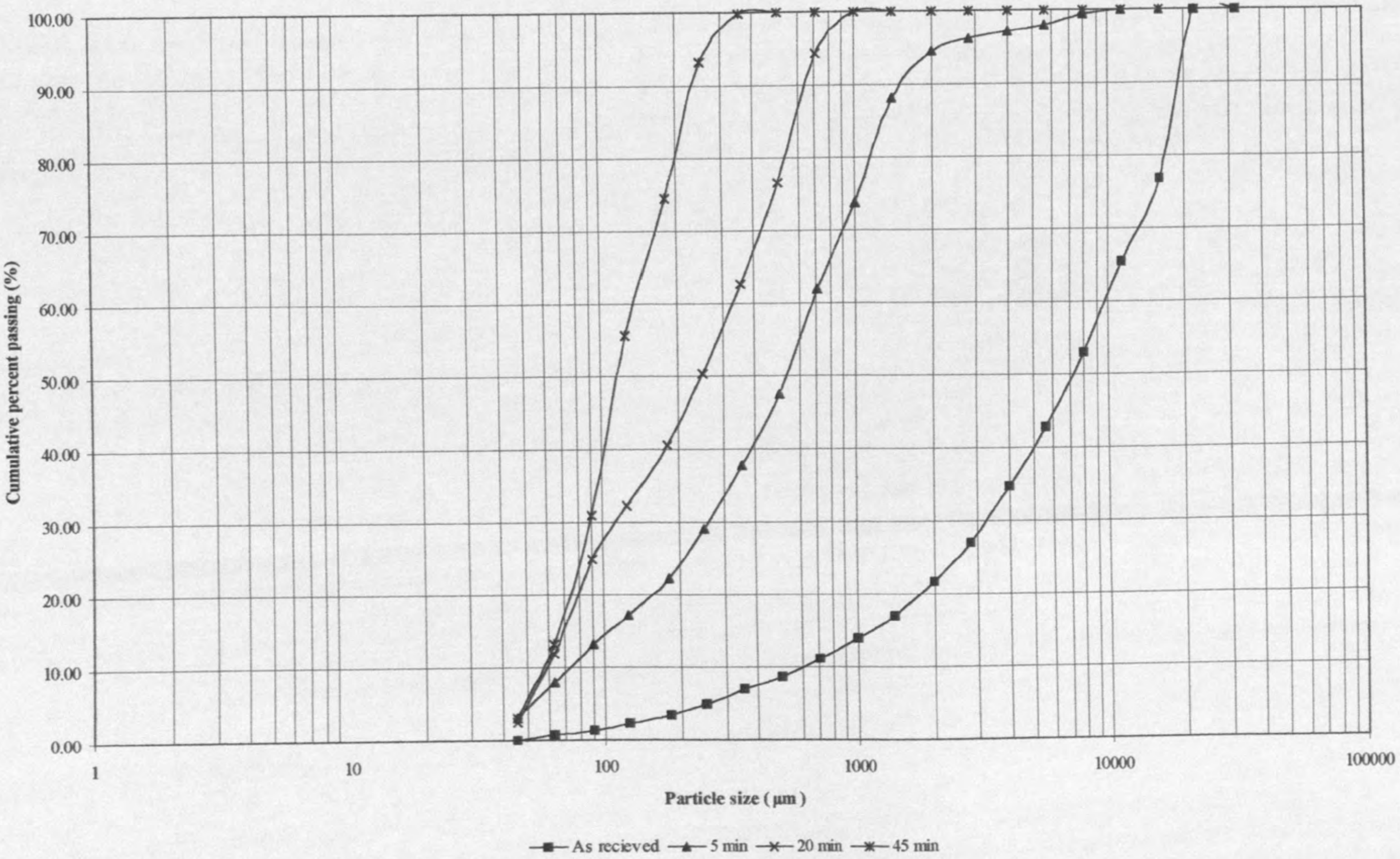
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-1 minute



P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-2 minute

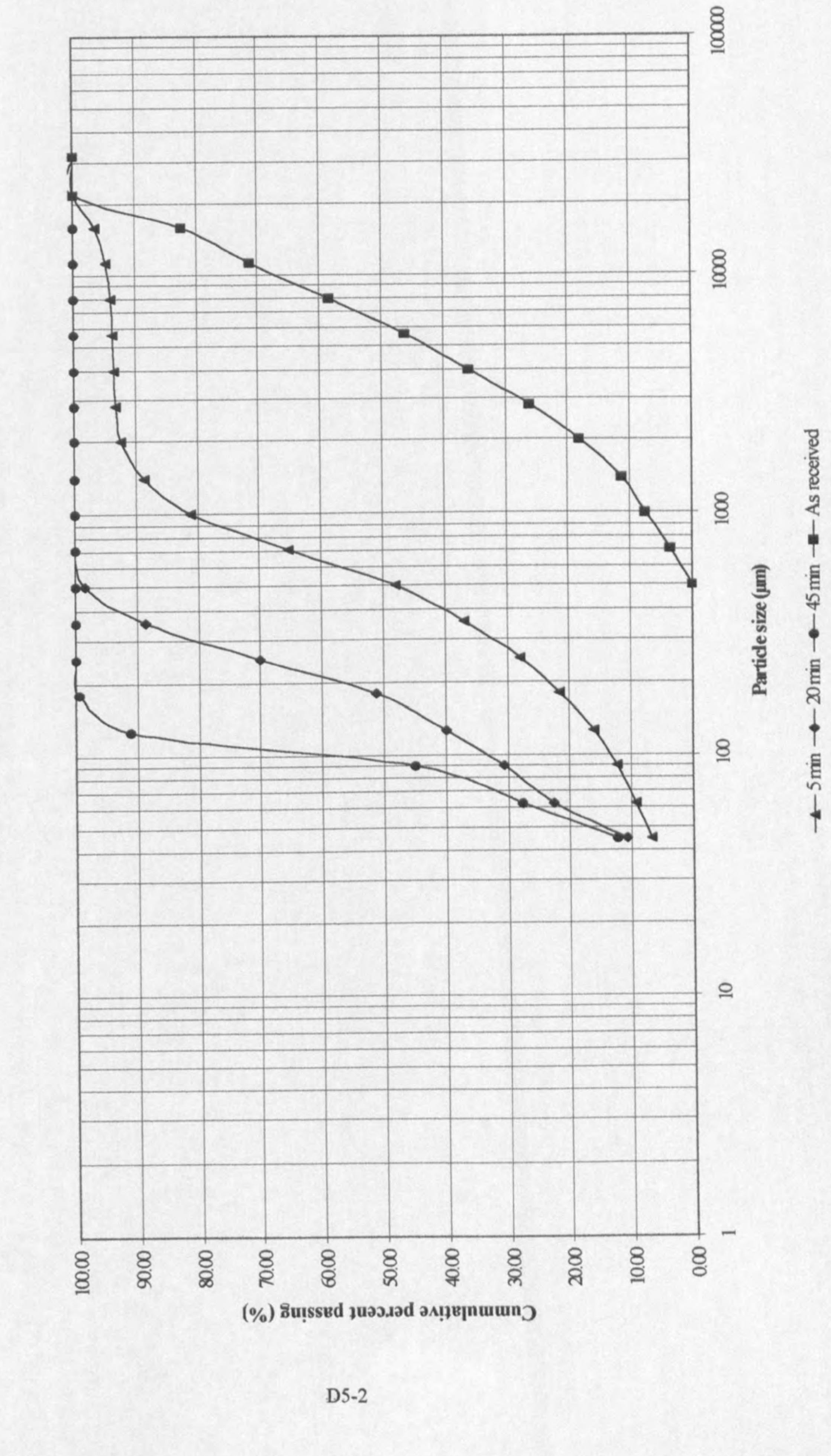


P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-3 minute

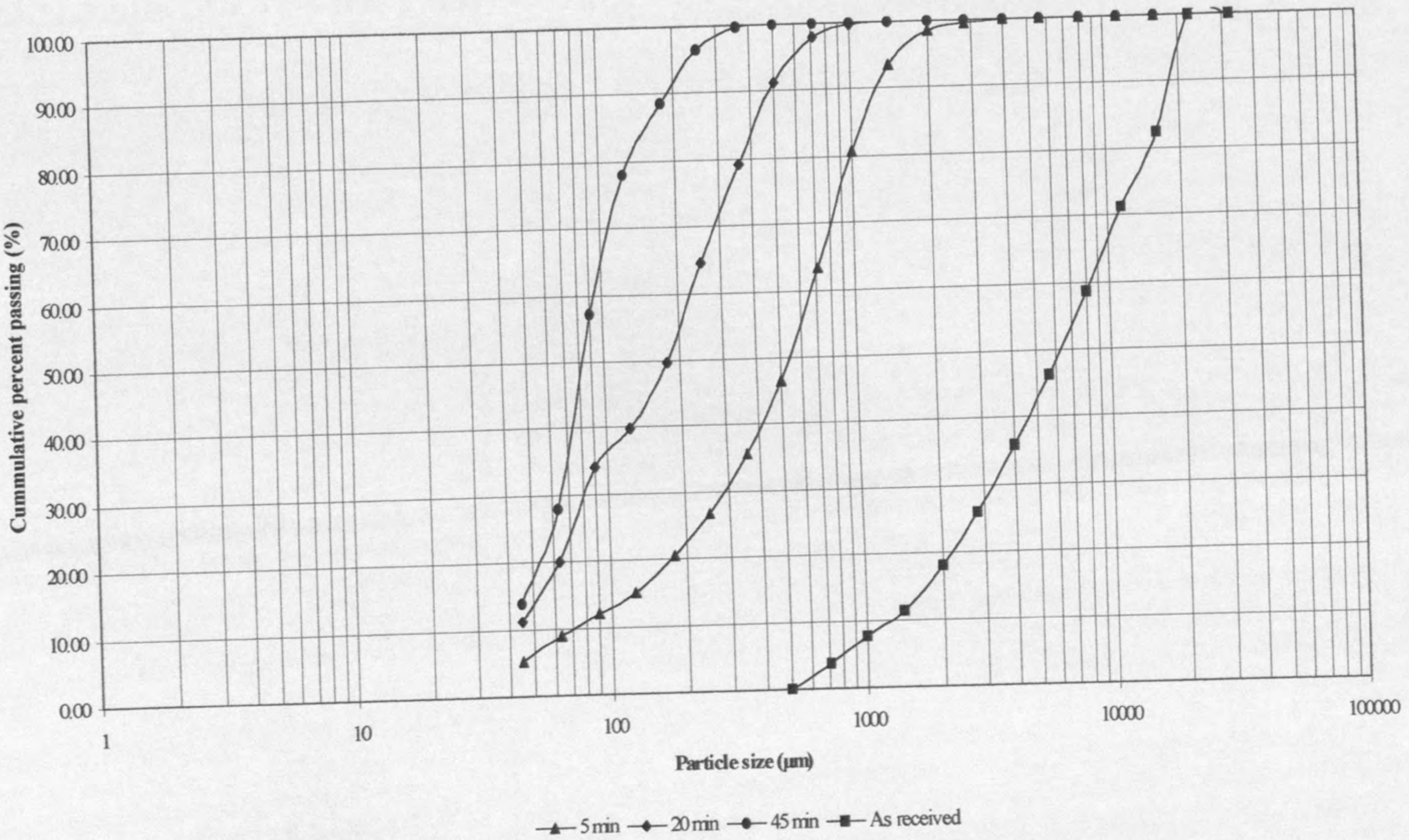


APPENDIX D5

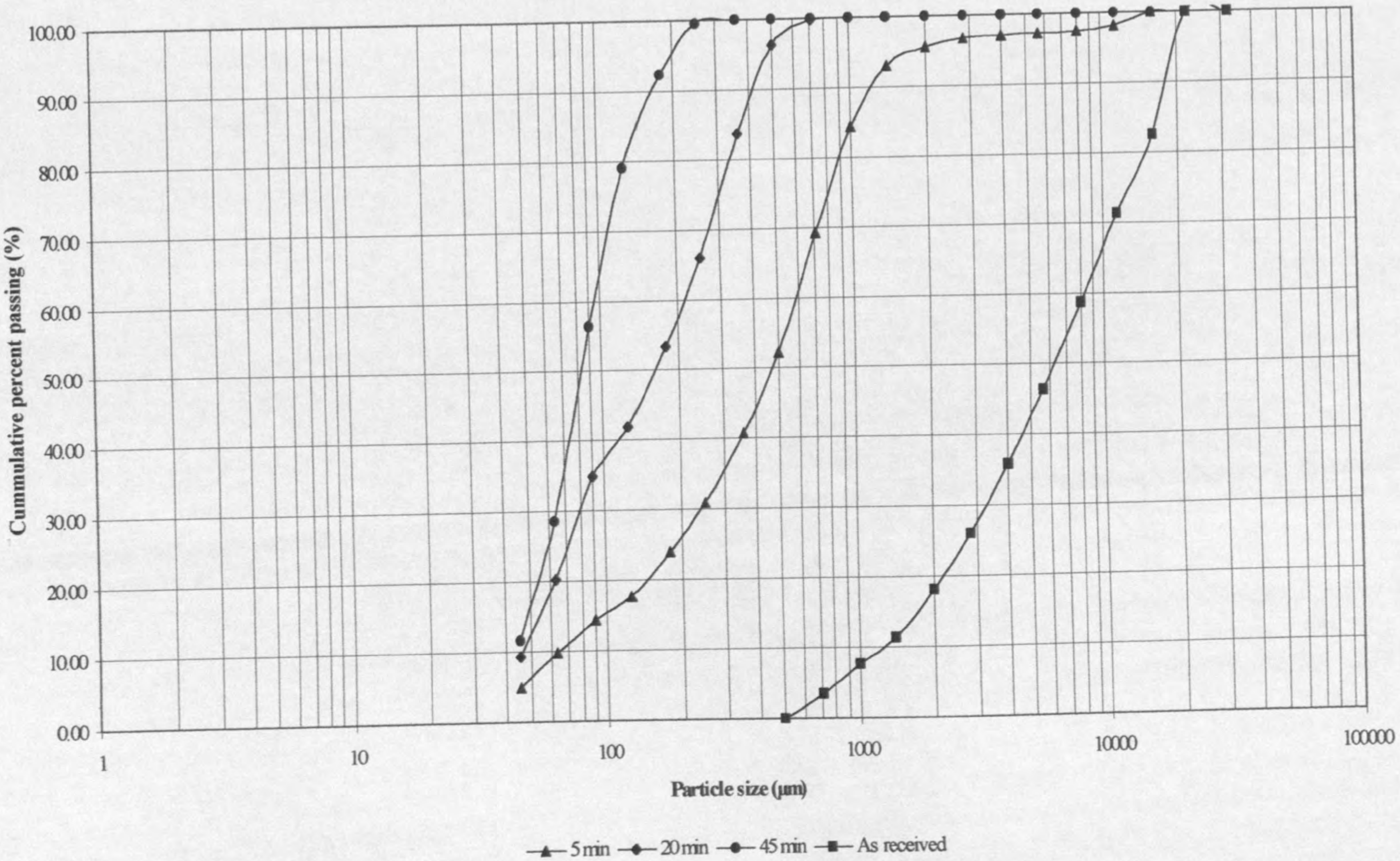
MICROWAVE TREATED COAL AT CONSTANT ENERGY INPUT-VARYING POWER AND EXPOSURE TIME ROD MILL SIZE DISTRIBUTIONS


P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-609s, 220kWh/t

P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-1.3kW, applied frequency-2.45GHz, exposure time-305s, 220kWh/t



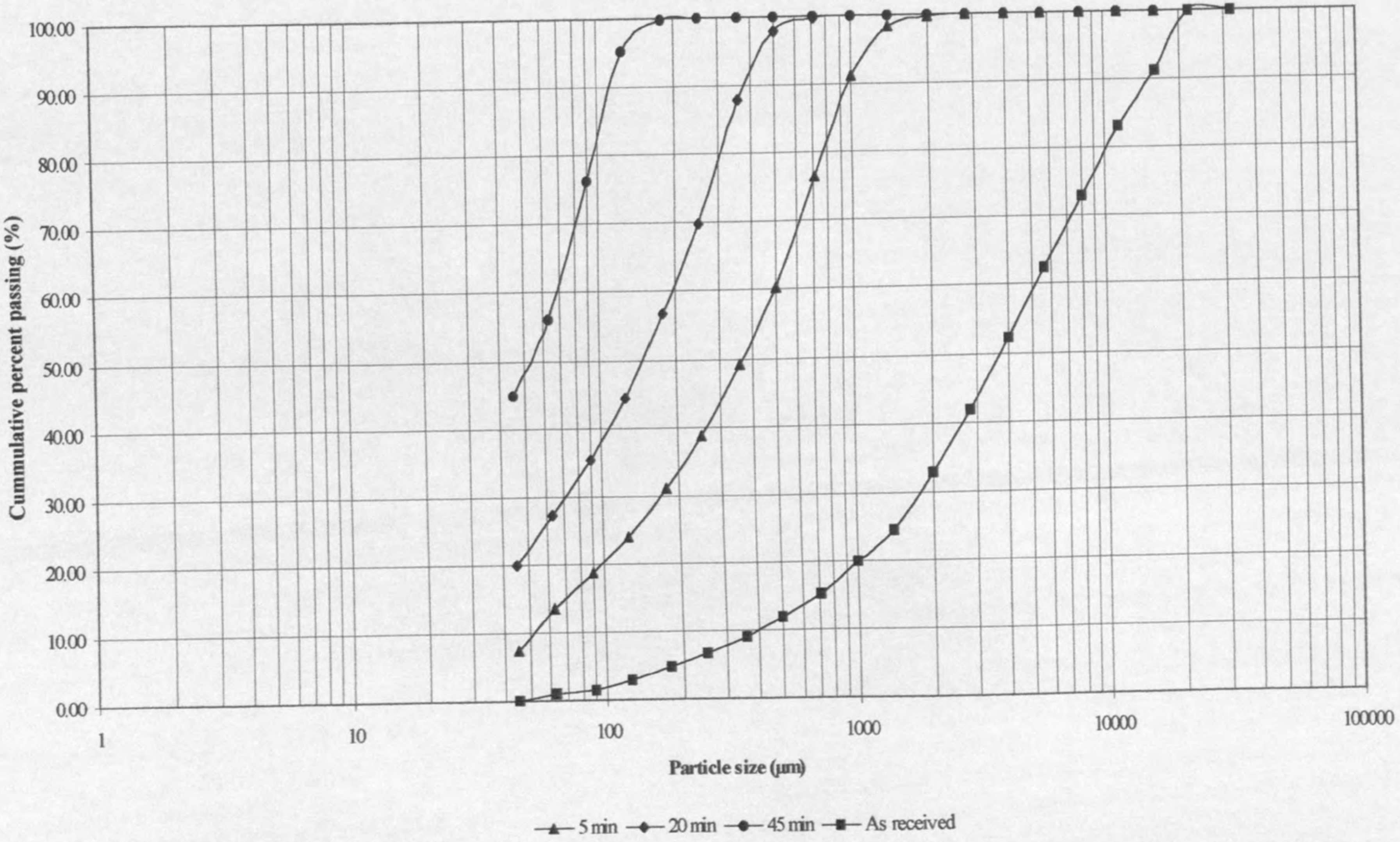
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-2.6kW, applied frequency-2.45GHz, exposure time-152s, 220kWh/t

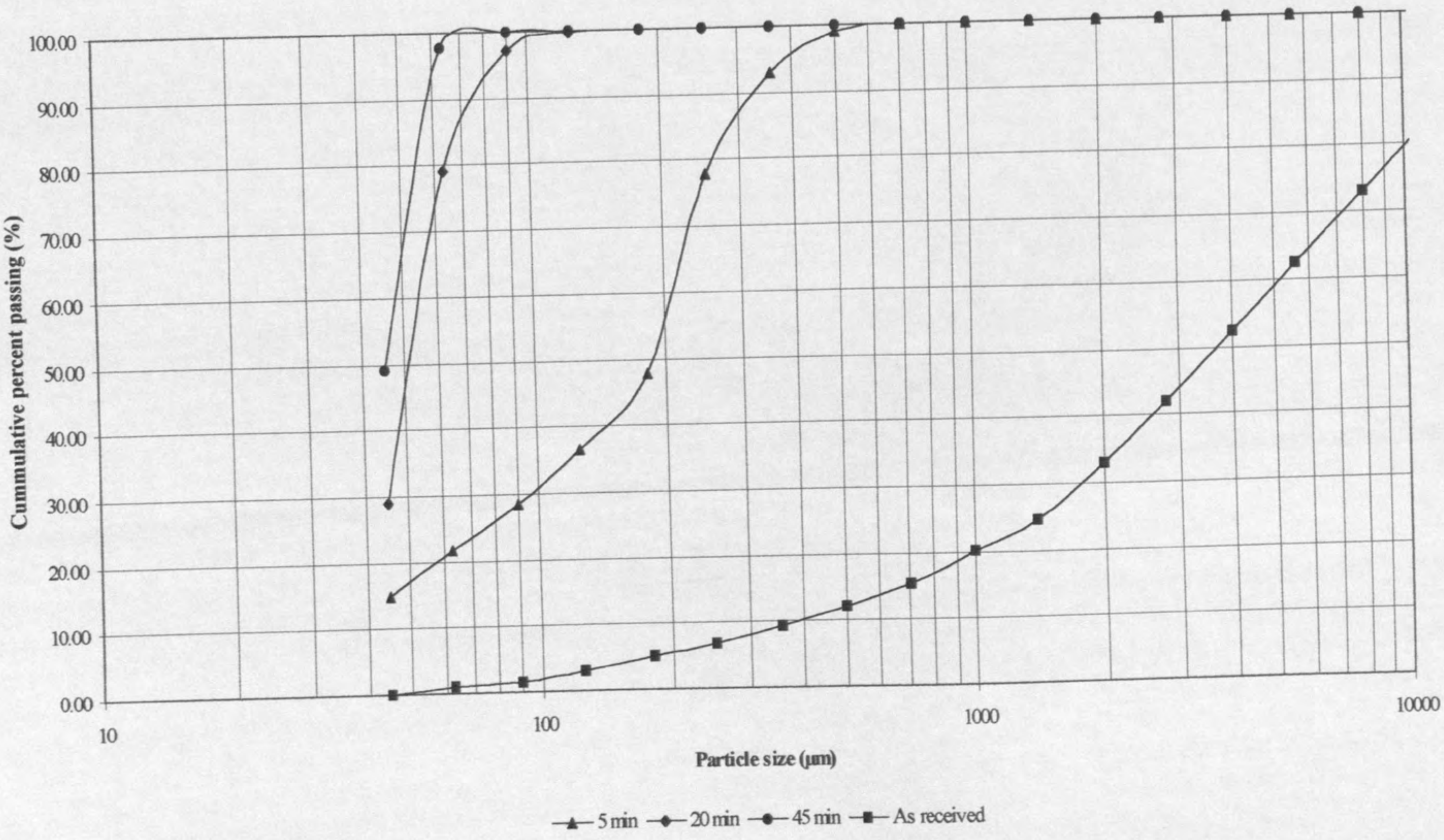


APPENDIX D6

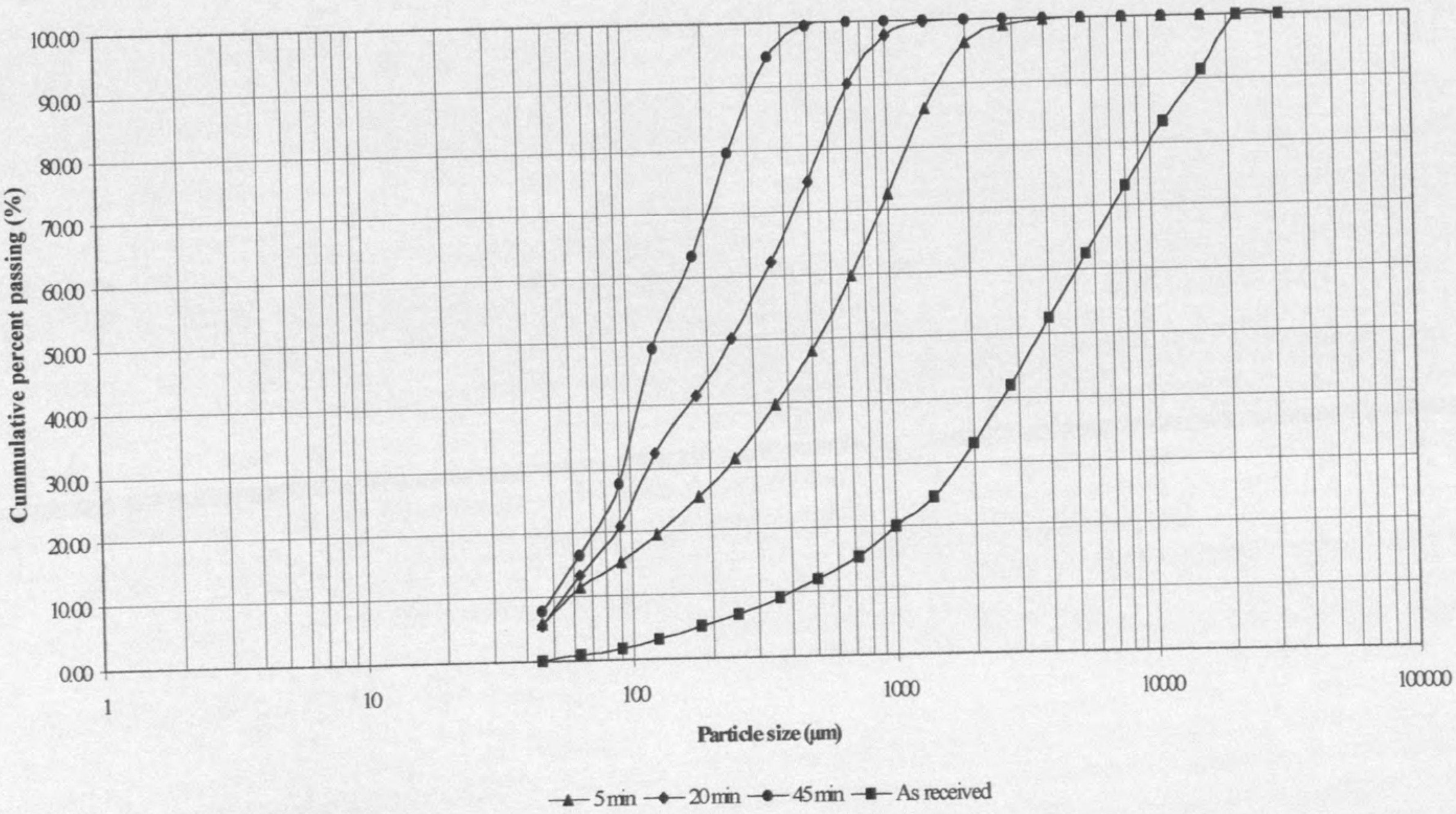
MICROWAVE TREATED COAL AT CONSTANT ENERGY INPUT VARYING SAMPLE LOAD ROD MILL SIZE DISTRIBUTIONS

P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes (173kWh/t)
milled as 500g



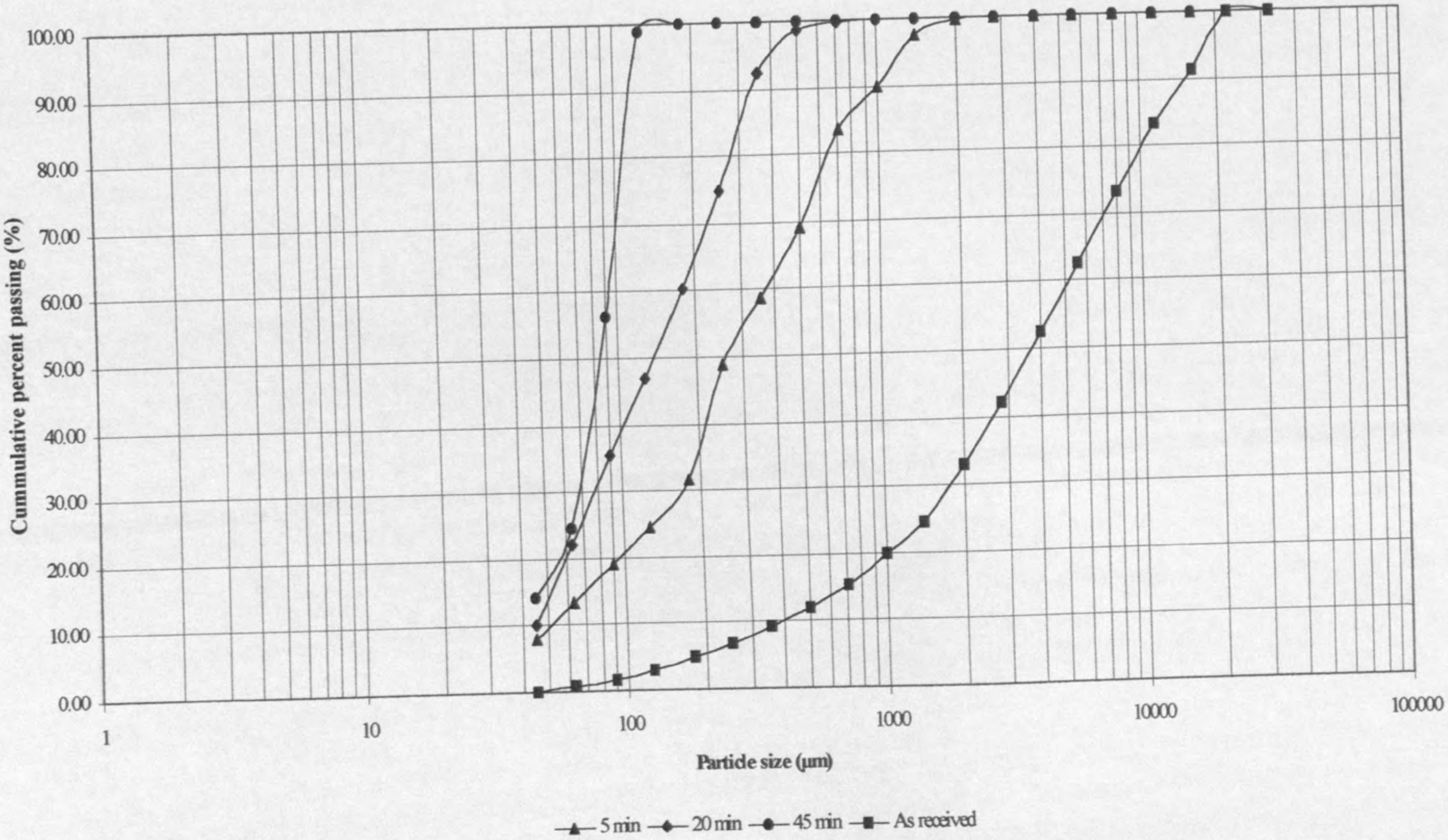


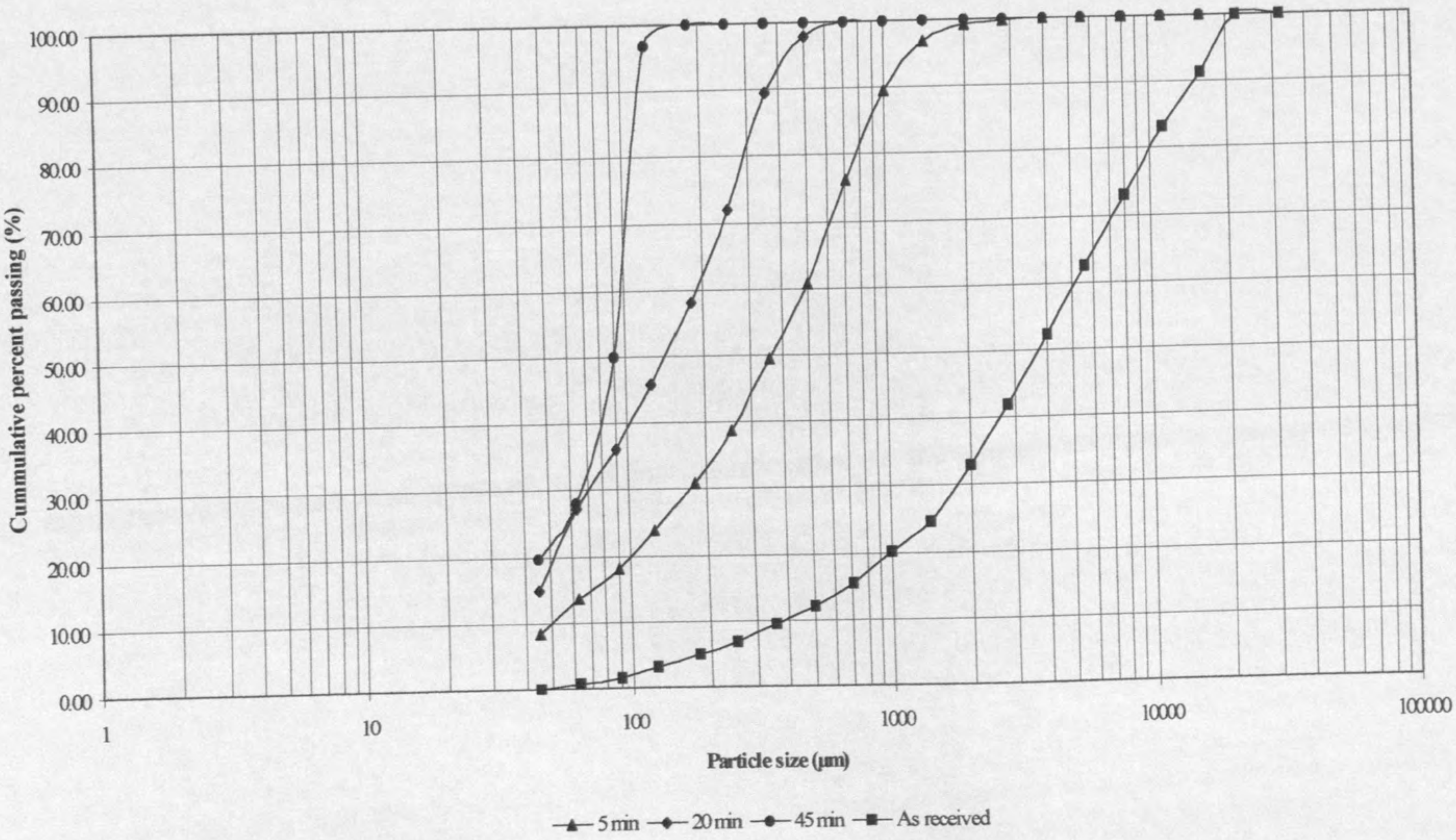
P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-250g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-4 minutes (173kWh/t)
milled as 250g



P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-1000g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-16 minutes (173kWh/t)
milled as 1000g

P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-250g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-4 minutes (173kWh/t)
milled as 500g (2 samples of 250g)

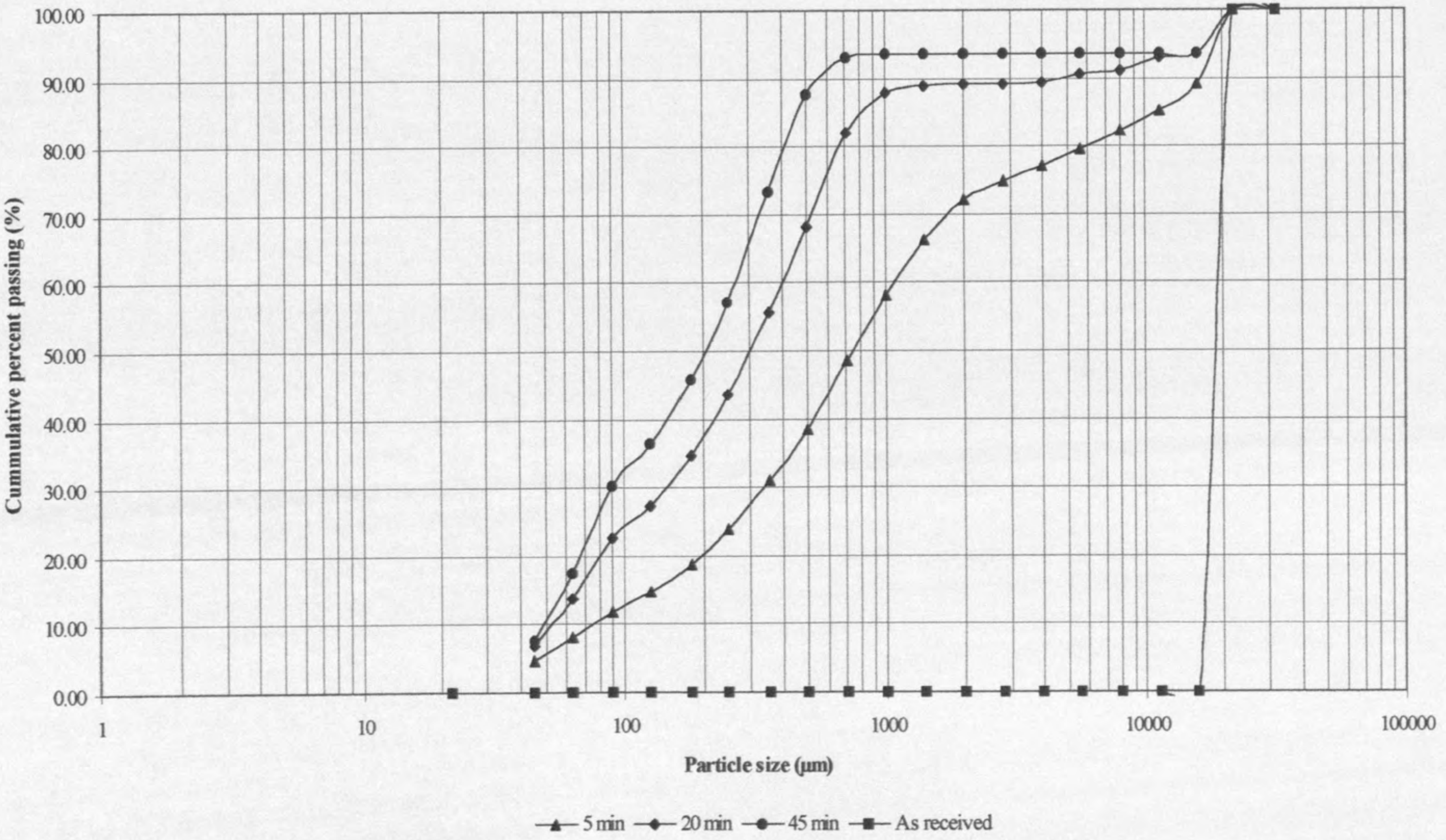




P-1 coal , rod mill, Number of rods-7, operating speed-100rpm, sample load-1000g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-16 minutes (173kWh/t)
milled as 500g (sample divided in 2)

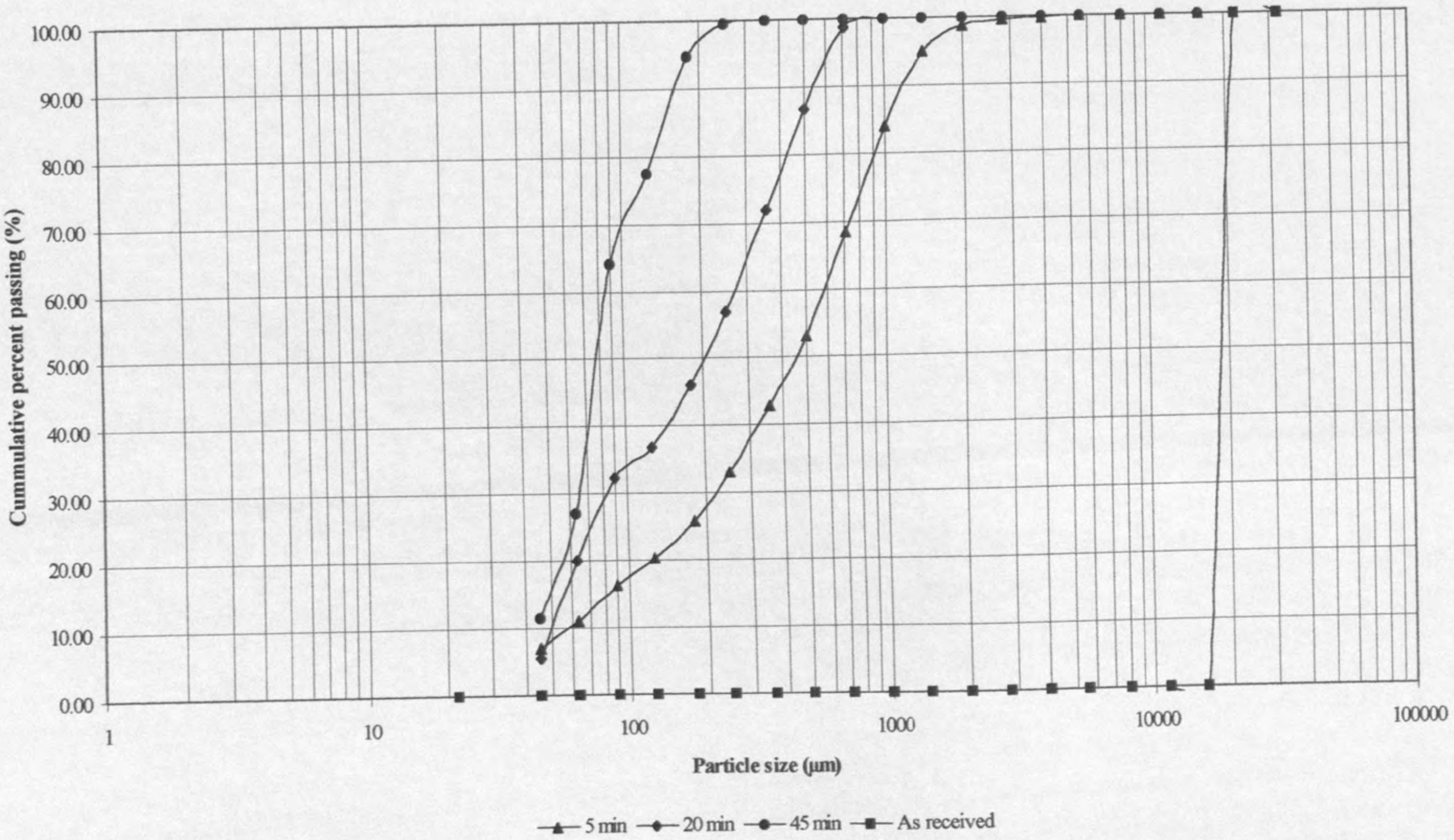
APPENDIX D7

MICROWAVE TREATED COAL AT CONSTANT ENERGY INPUT PARTICLE SIZE RANGE TESTS ROD MILL SIZE DISTRIBUTIONS

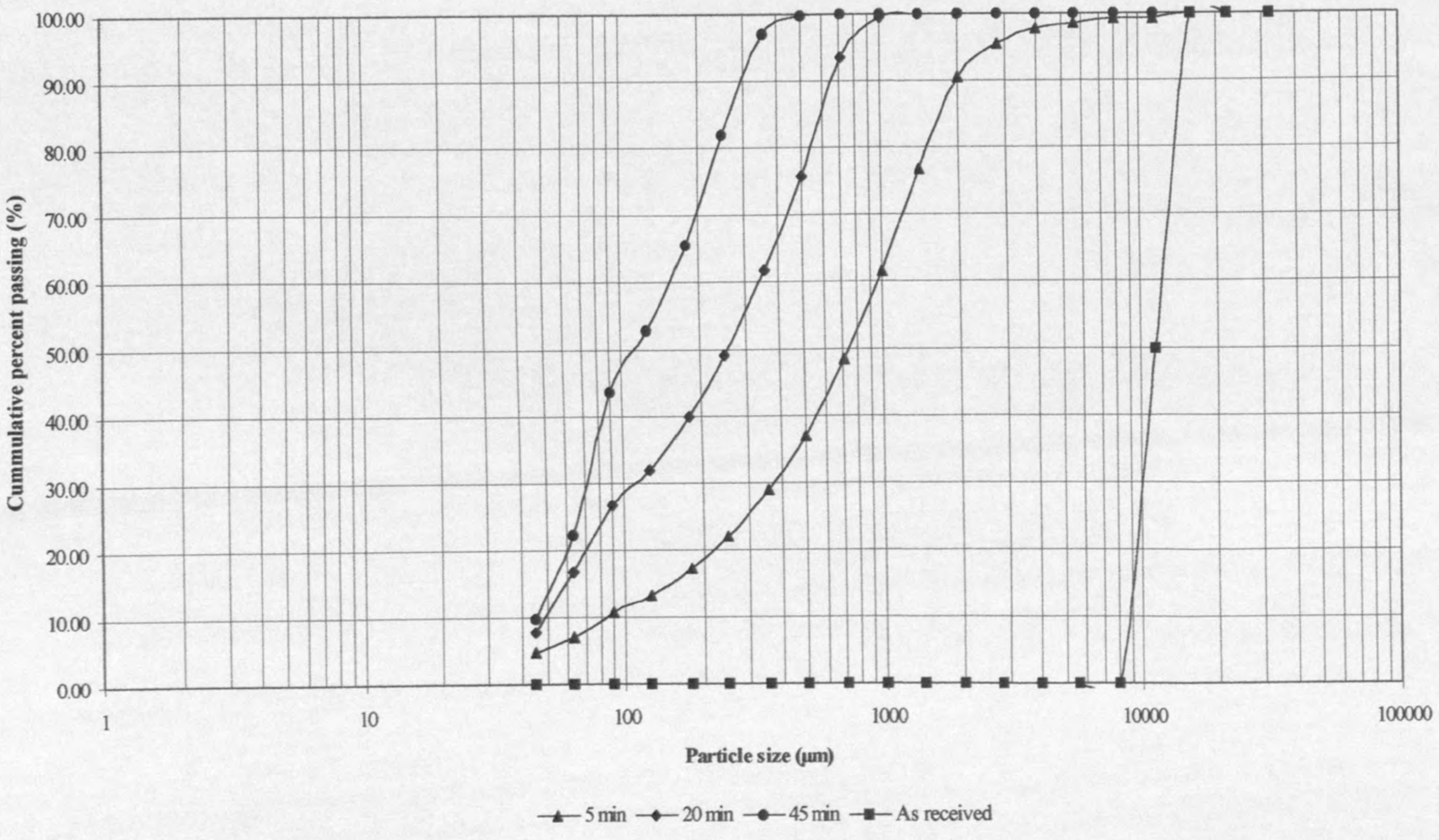


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range >16.0mm

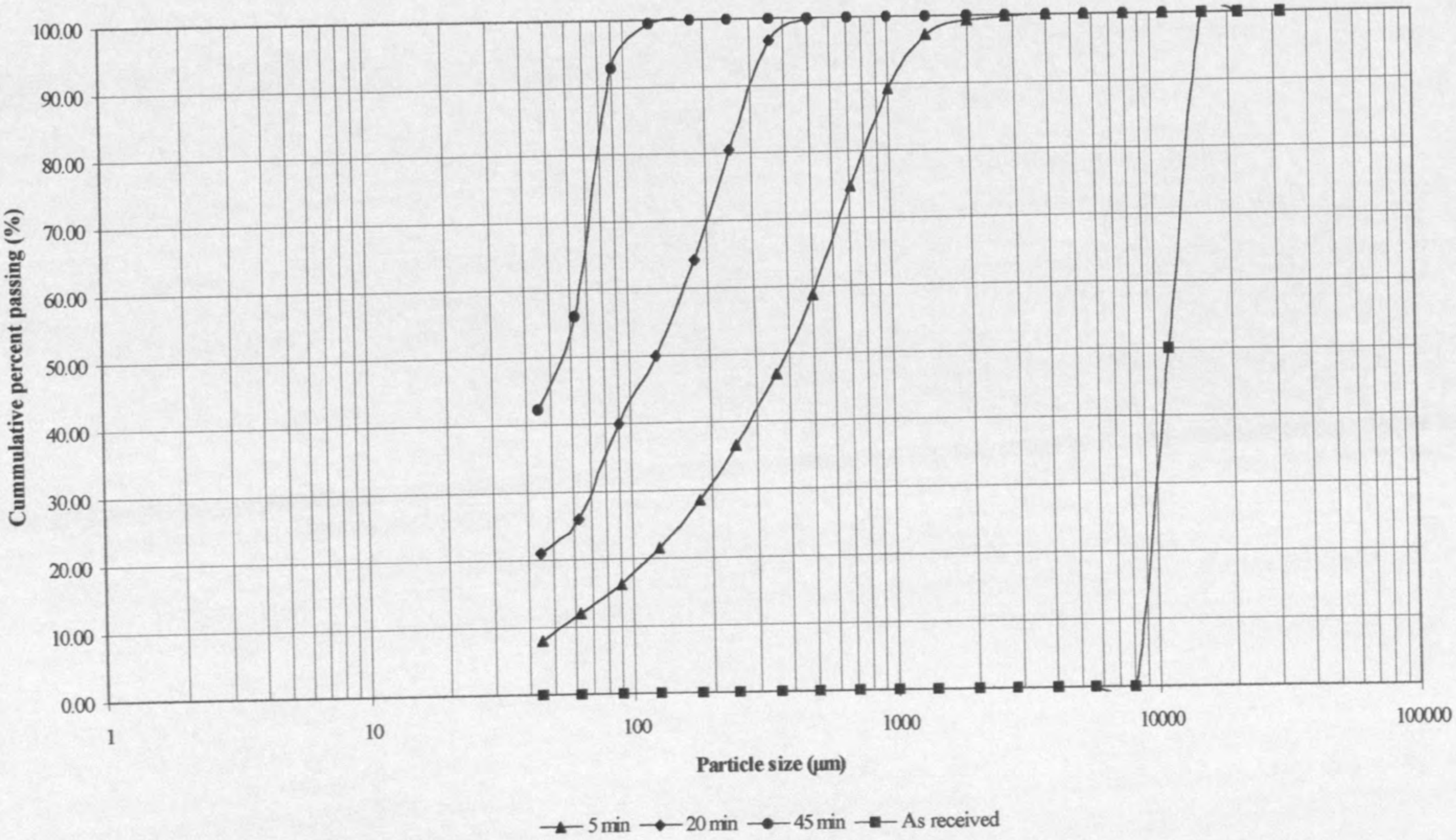
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range >16.0mm



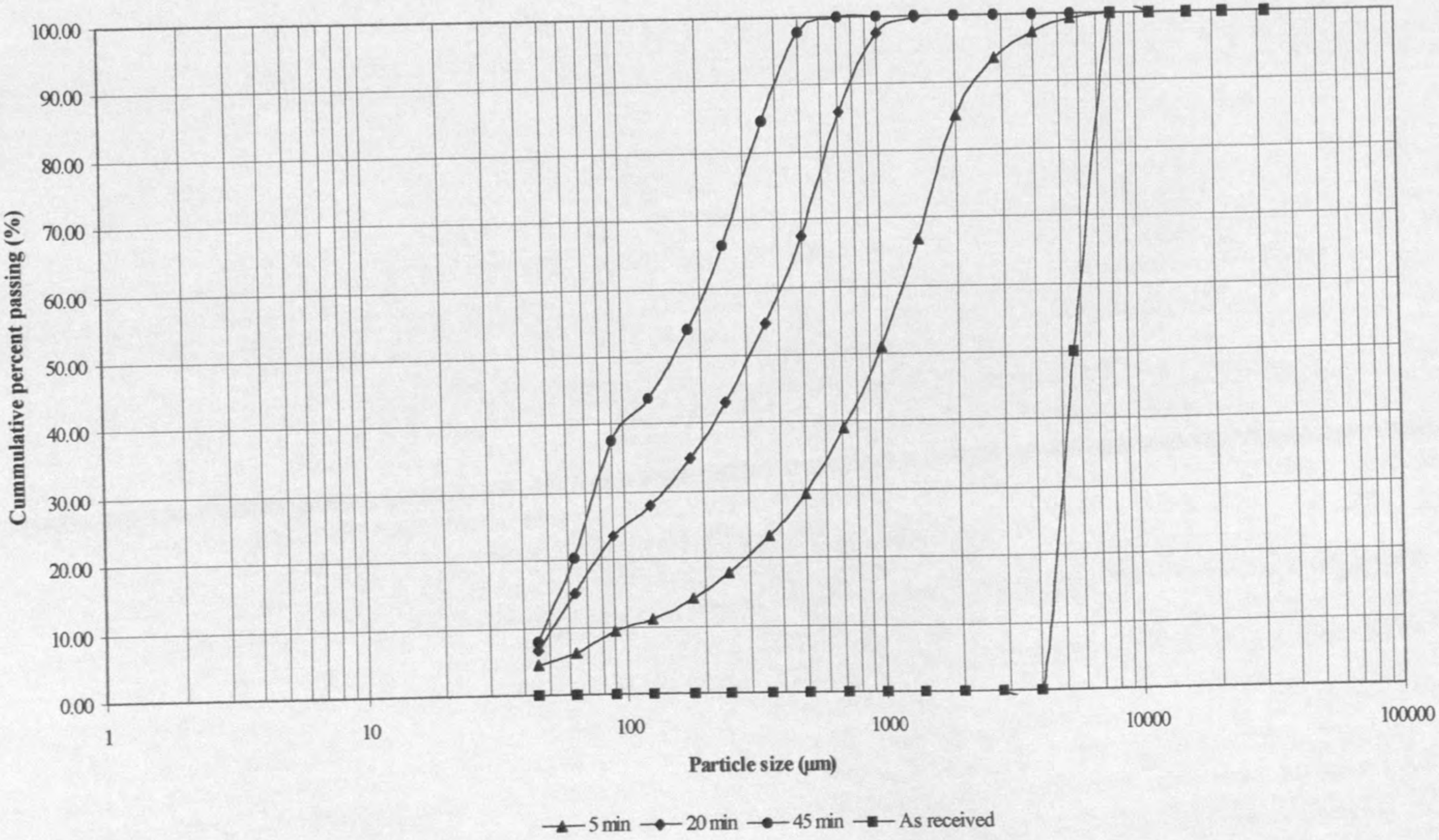
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -16.0 +8.0mm

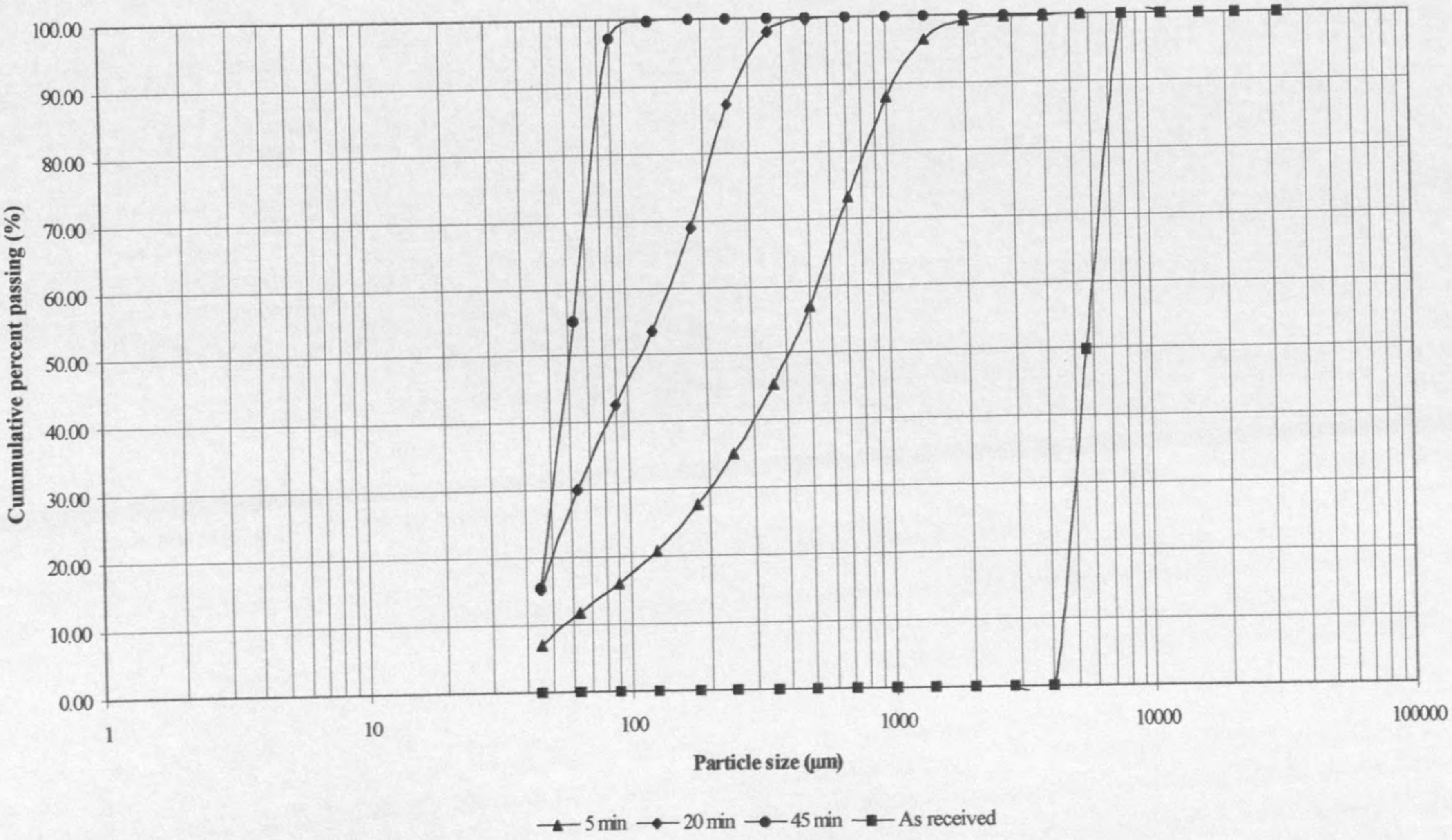


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -16.0 +8.0mm

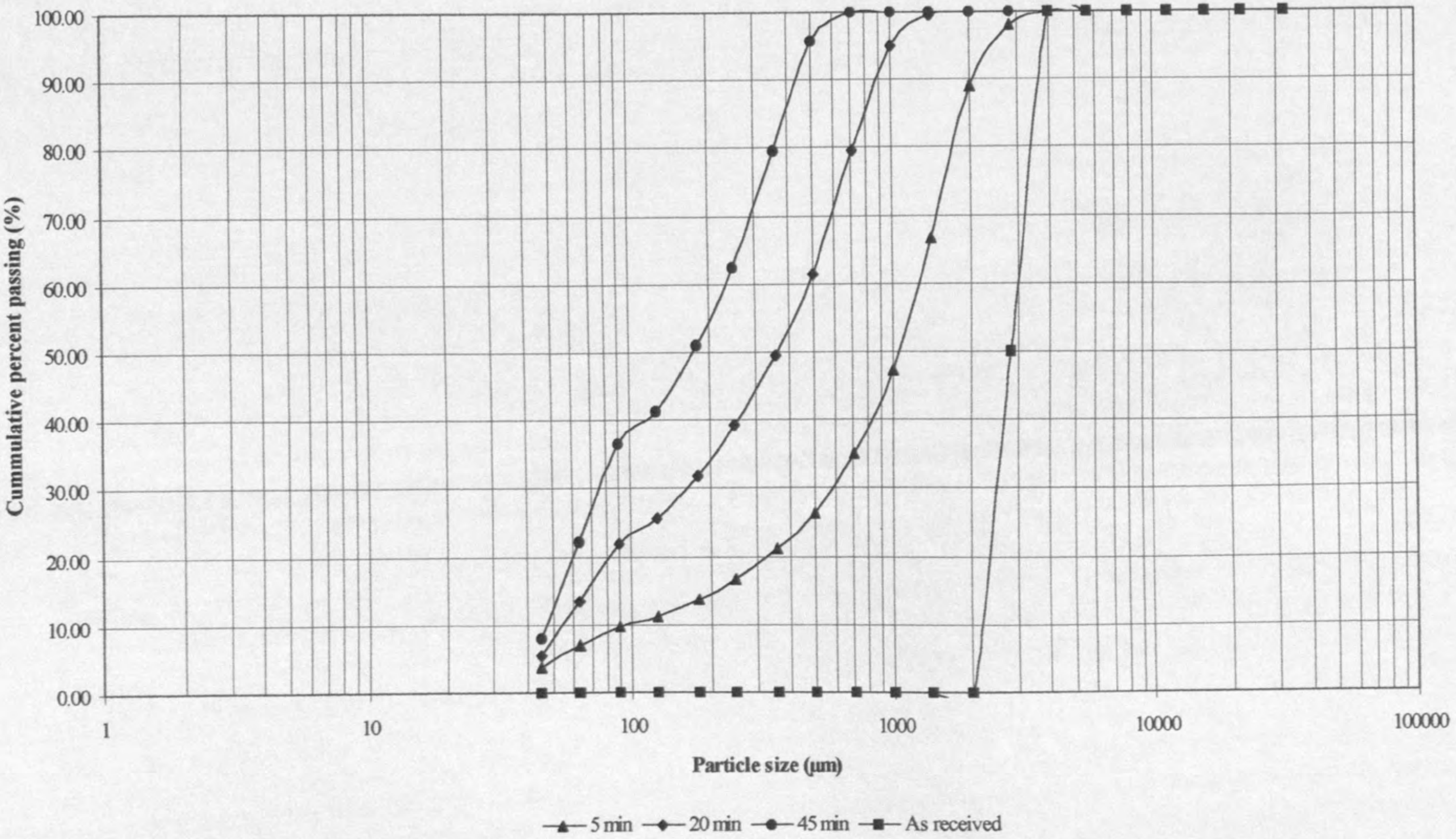


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -8.0 +4.0mm

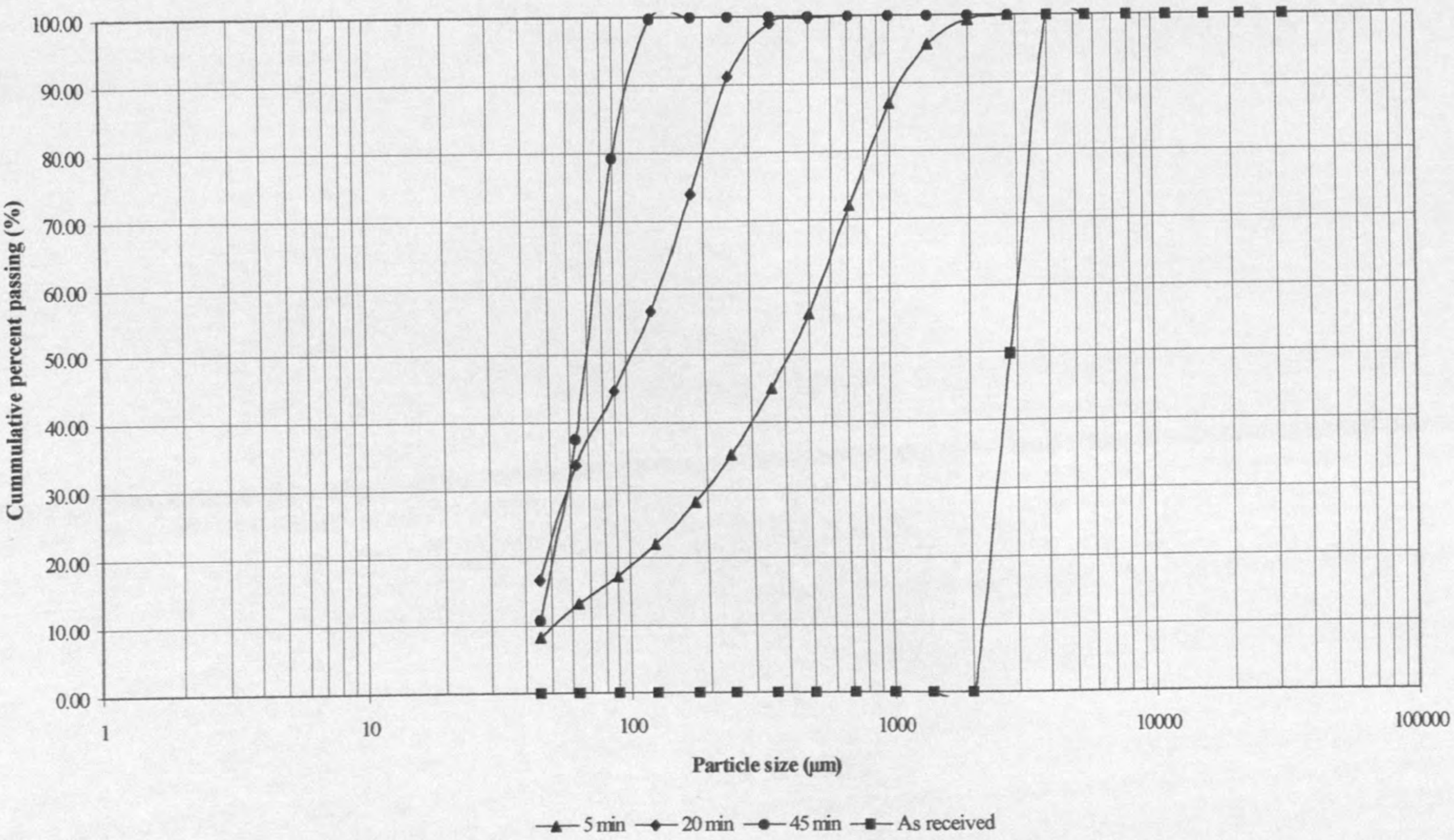



P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -8.0 +4.0mm

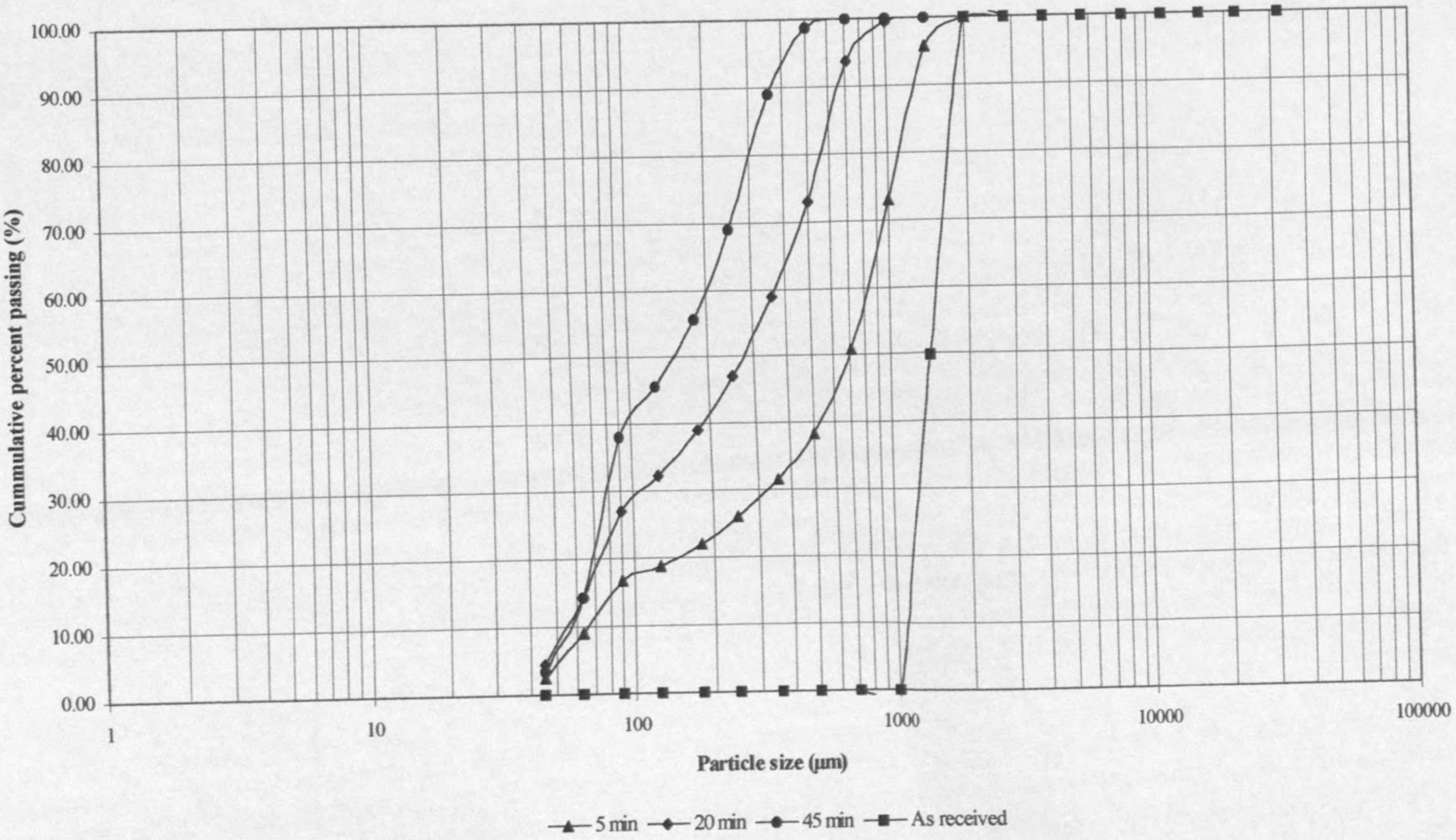
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -4.0 +2.0mm

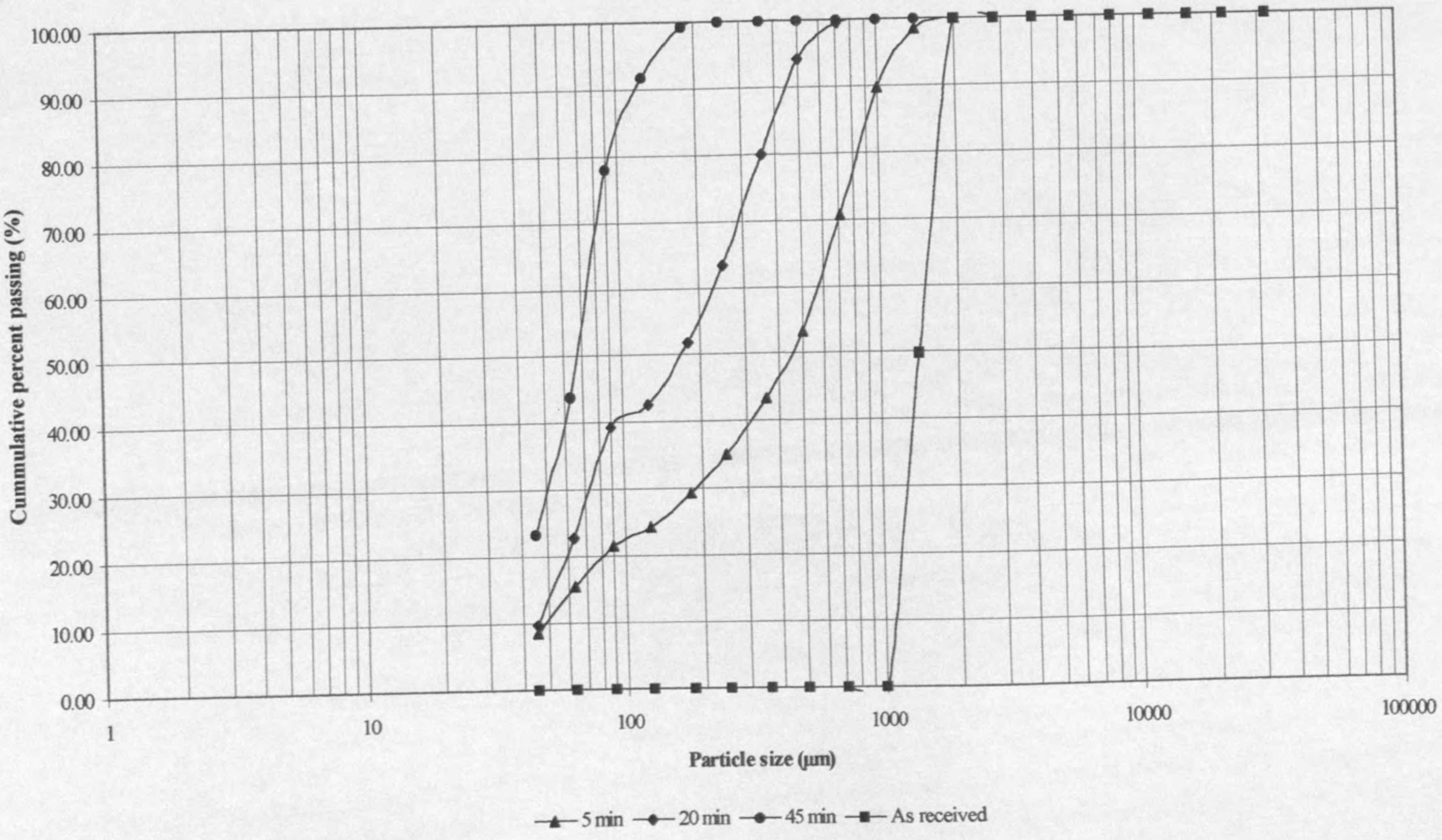


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -4.0 +2.0mm

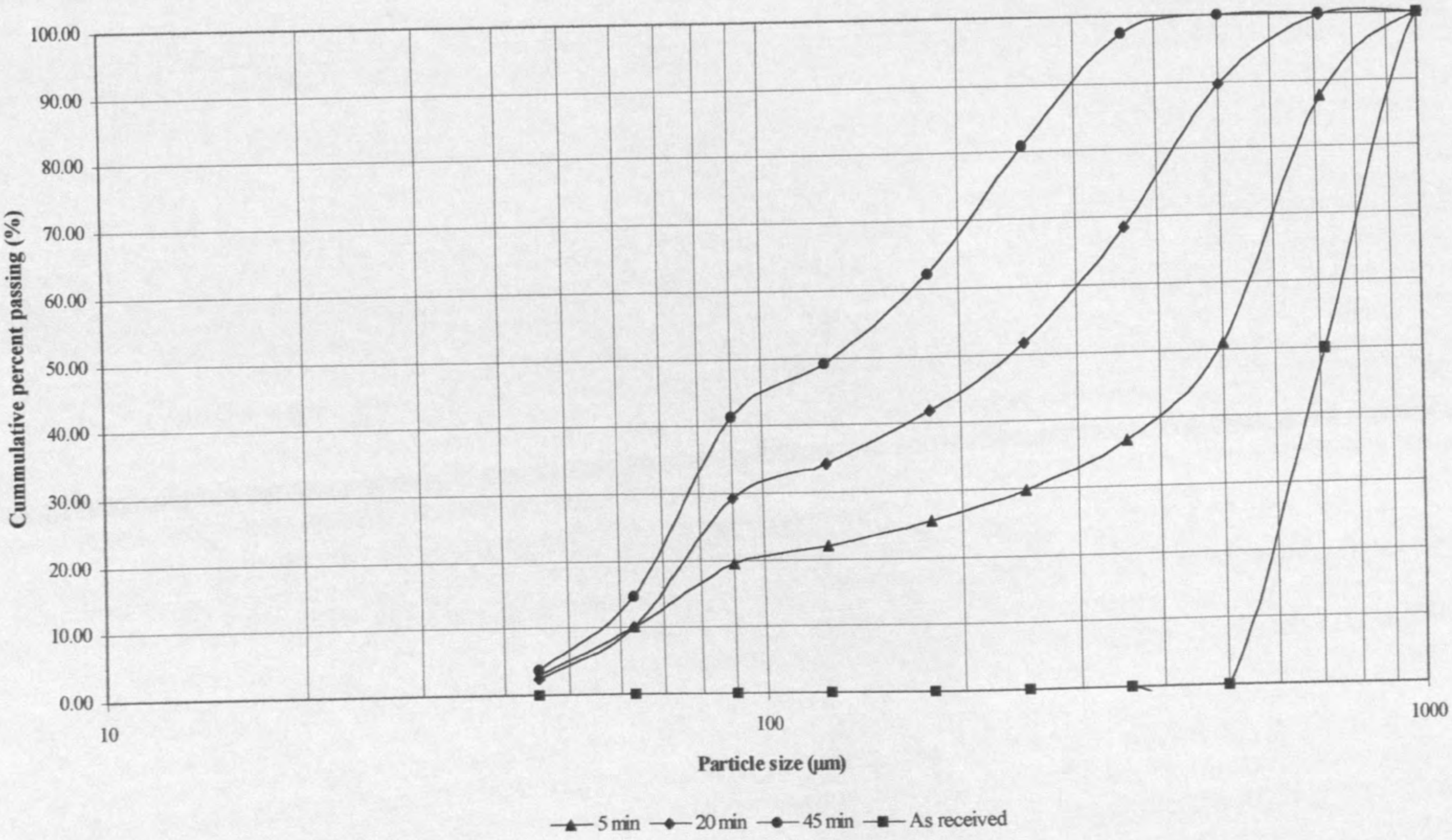


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -2.0 +1.0mm

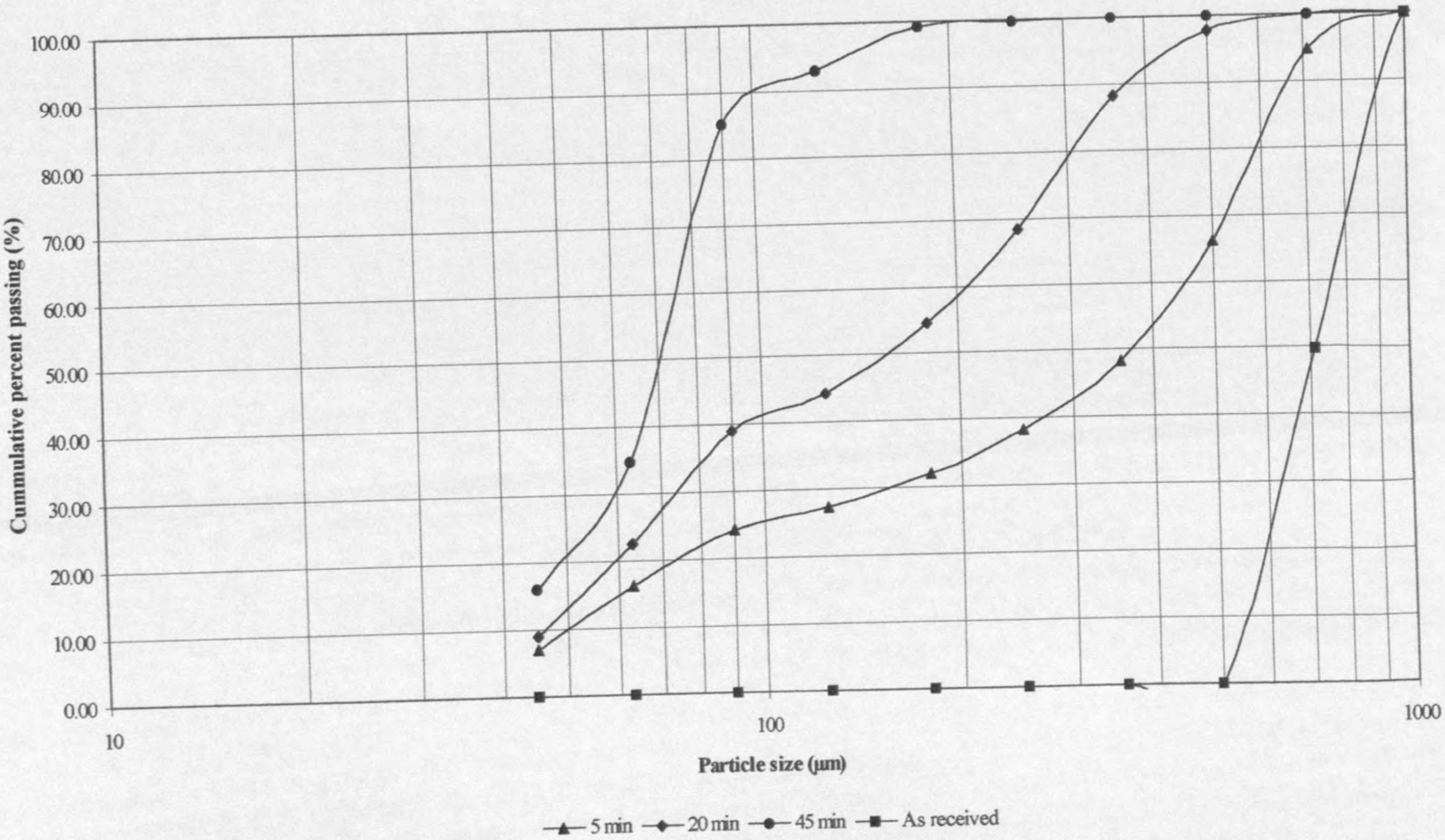



P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -2.0 +1.0mm

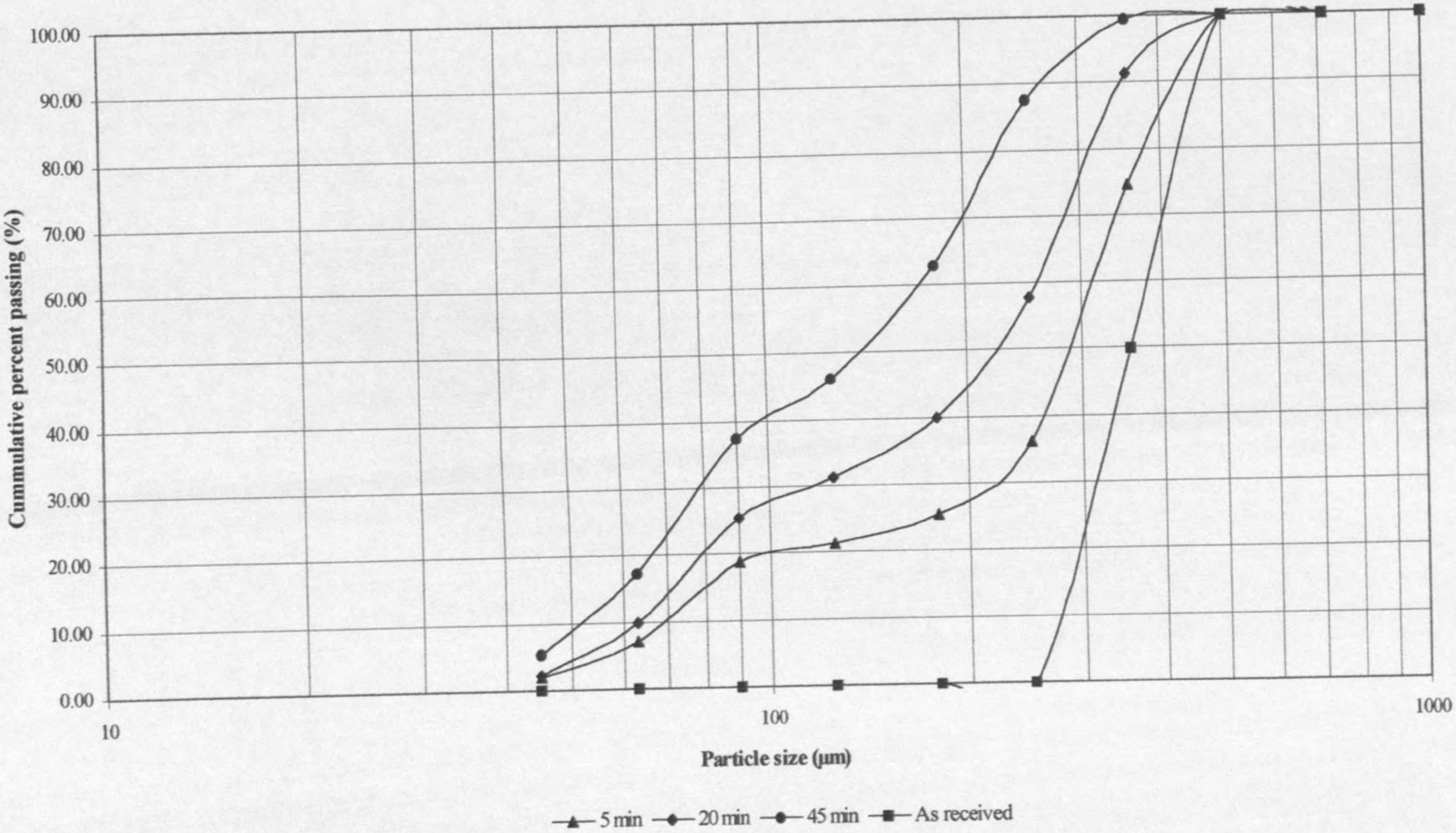
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -1.0 +0.5mm



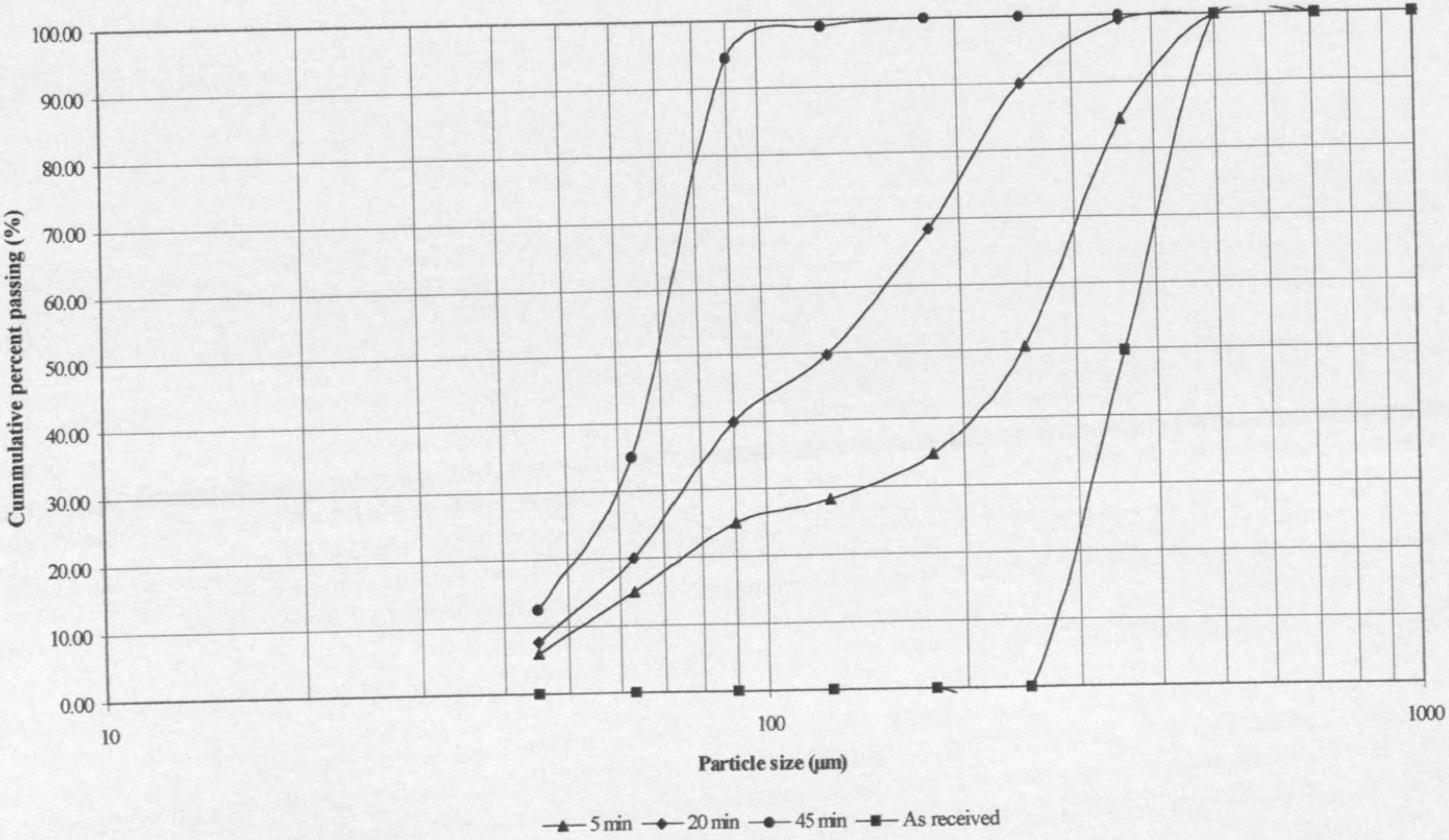
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -1.0 +0.5mm

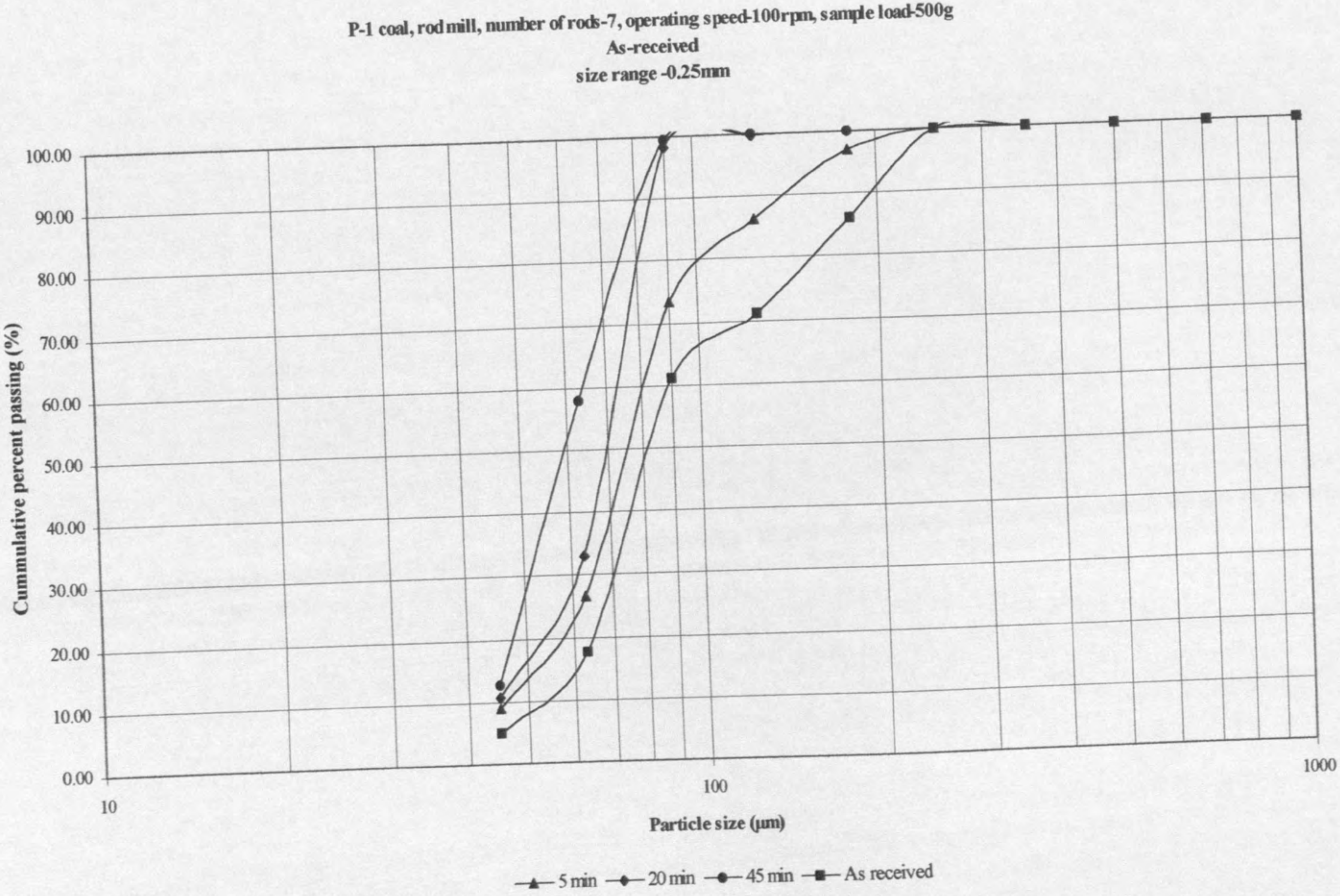


P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -0.5 +0.25mm

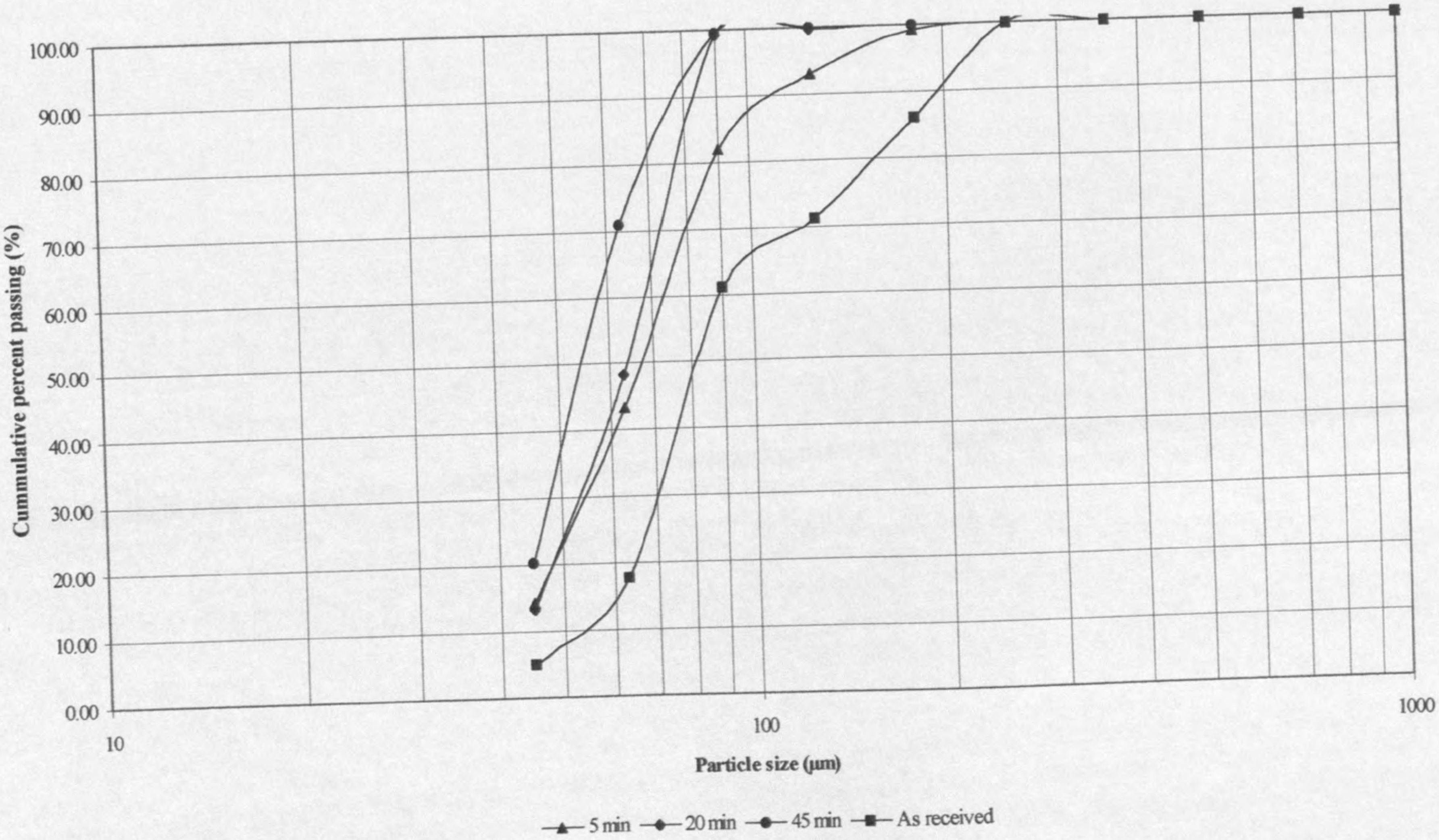


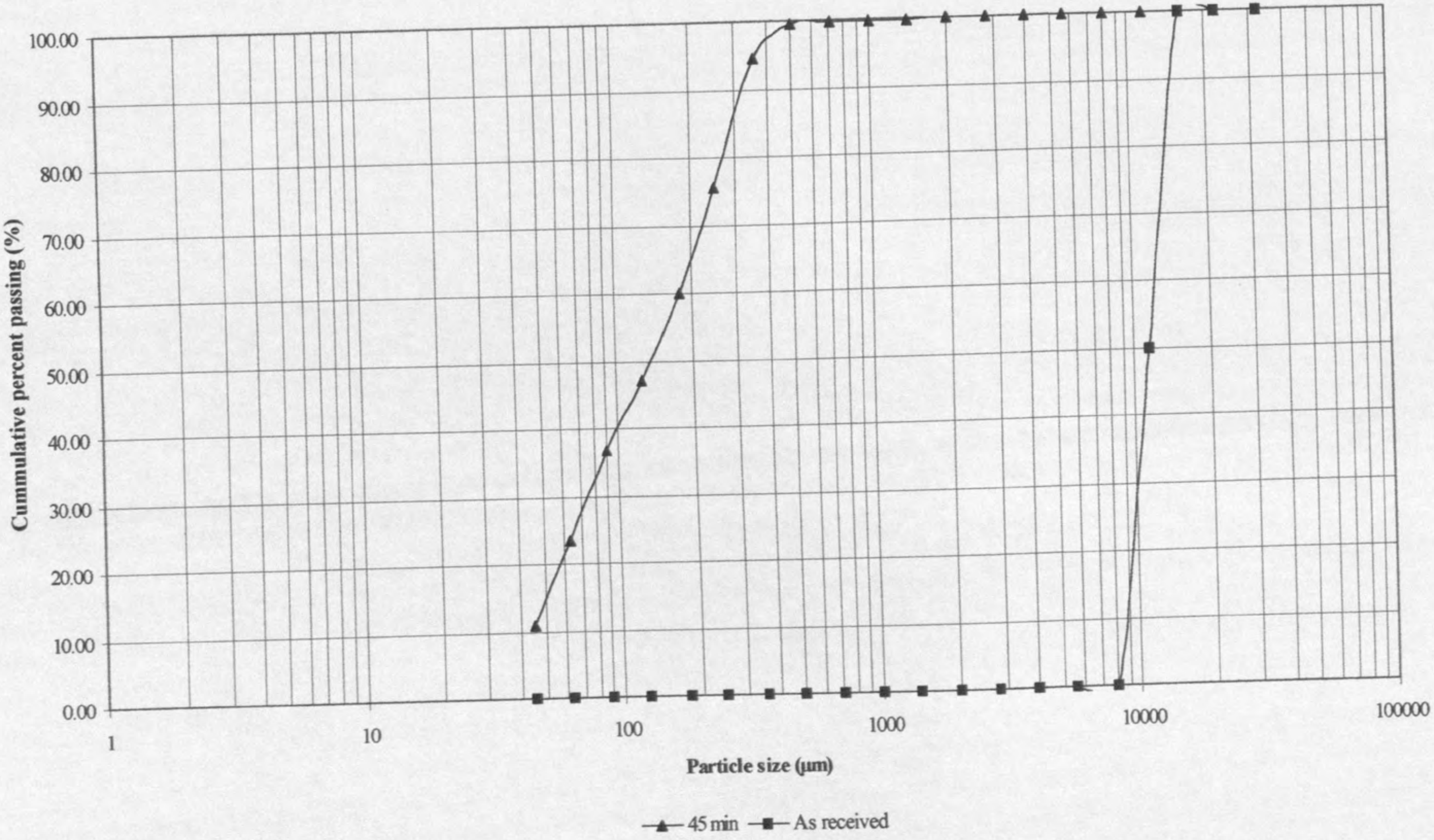
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -0.5 +0.25mm




P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -0.25mm
Cummulative percent passing (%)
100.00
90.00
80.00
70.00
60.00
50.00
40.00
30.00
20.00
10.00
0.00
10
100
1000
Particle size (µm)
5 min
20 min
45 min
As received

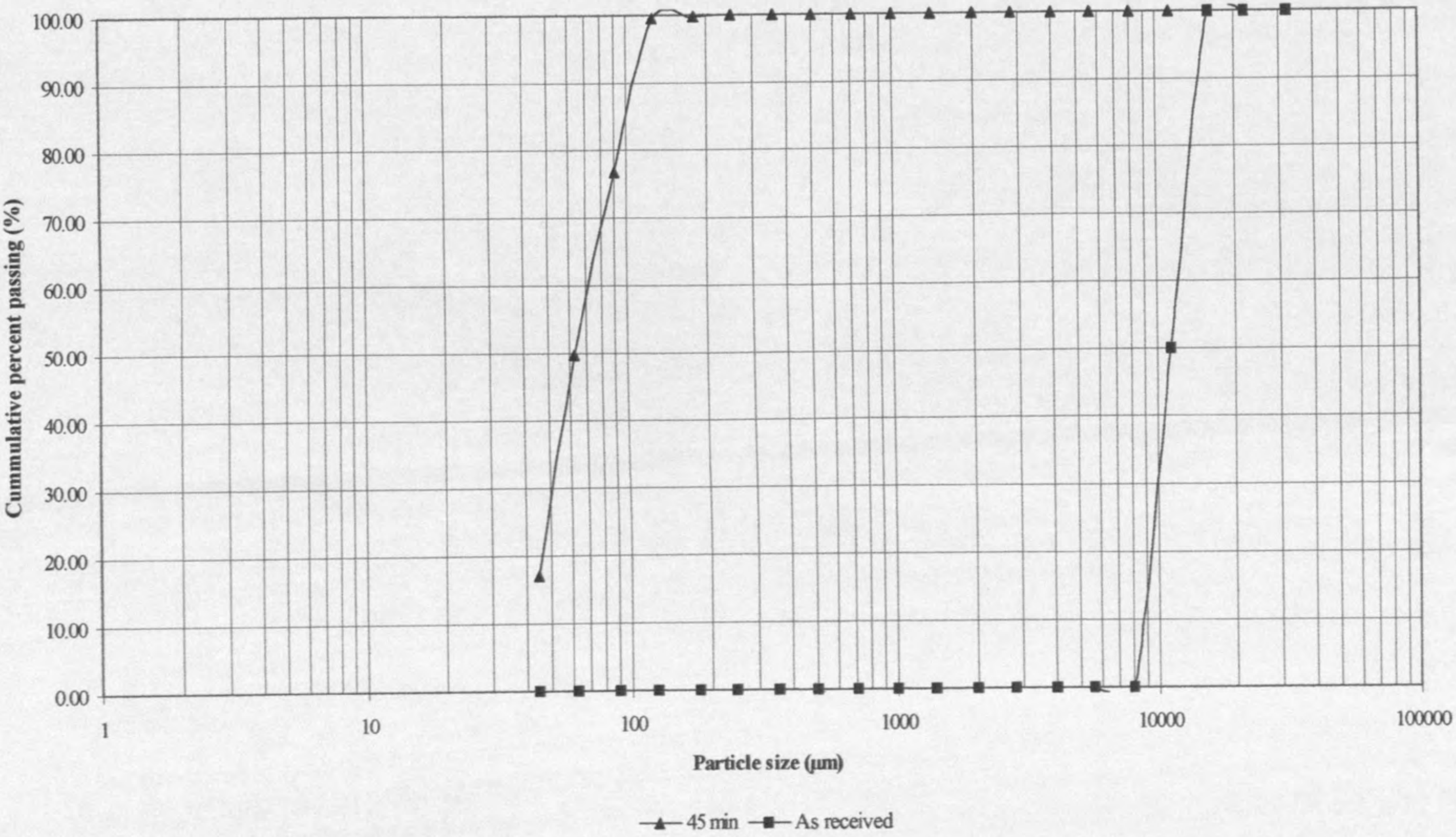
P-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -0.25mm



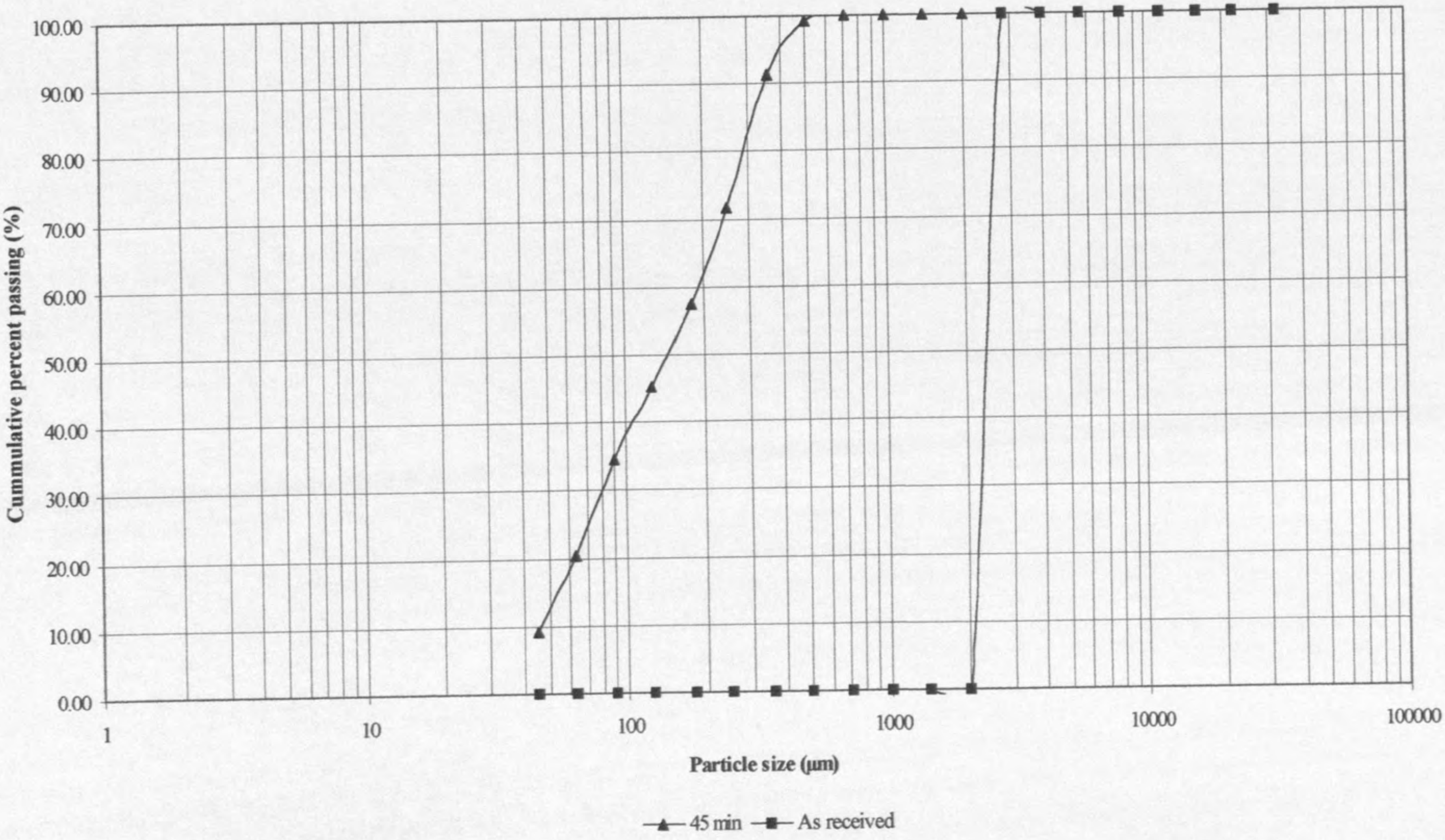


P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -16.0 +8.0mm

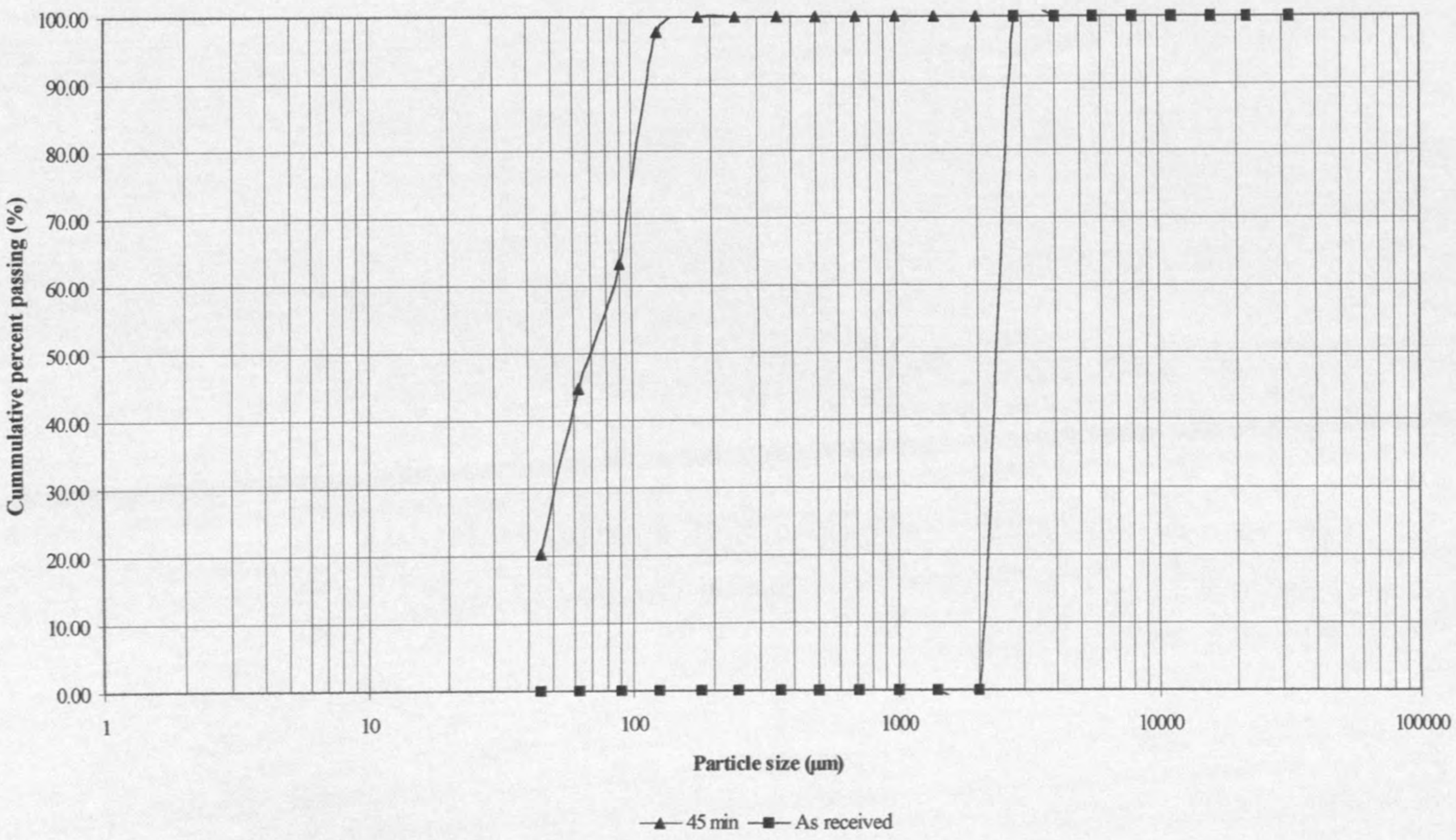
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -16.0 +8.0mm

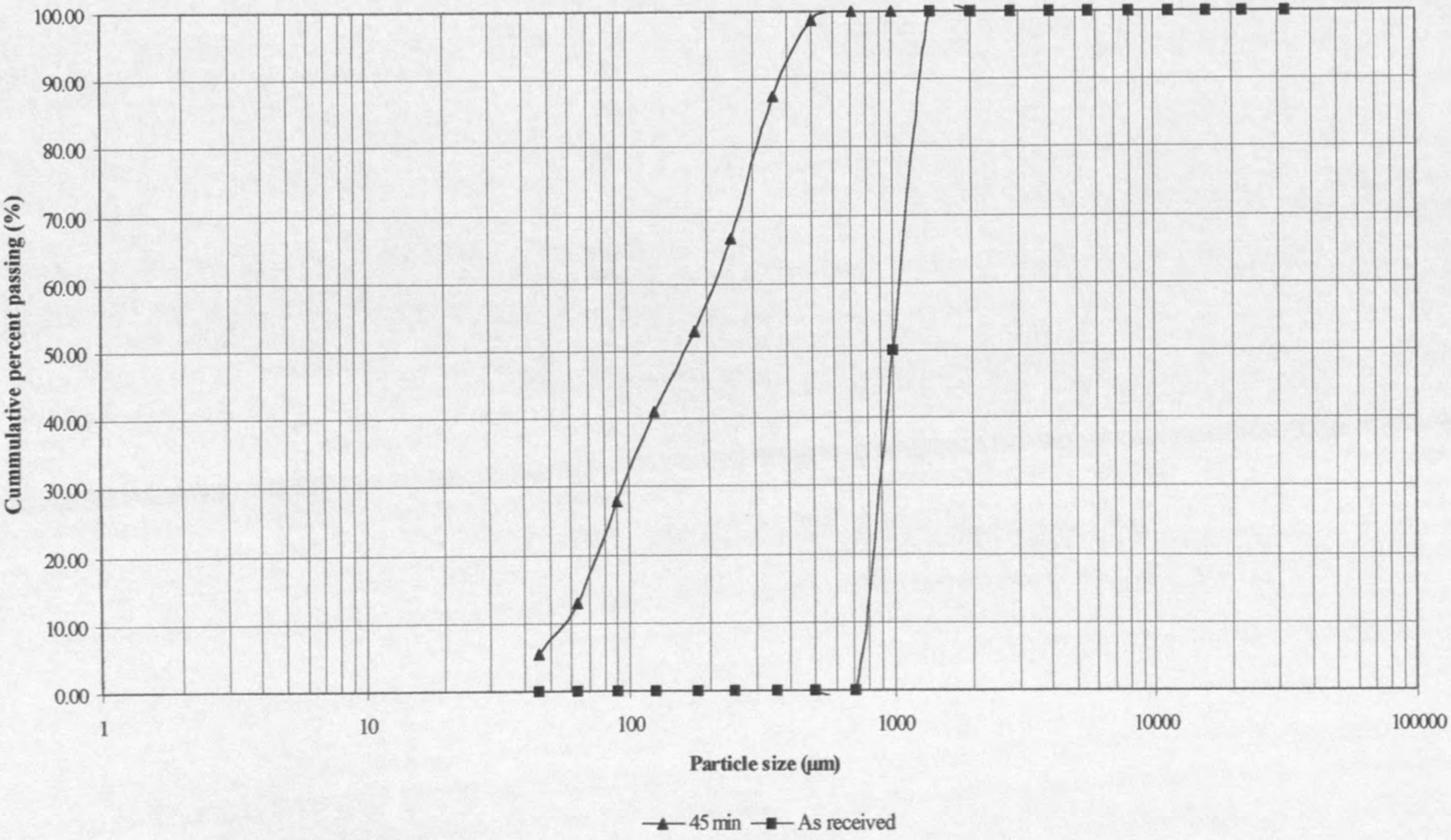


P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -2.8 +2.0mm



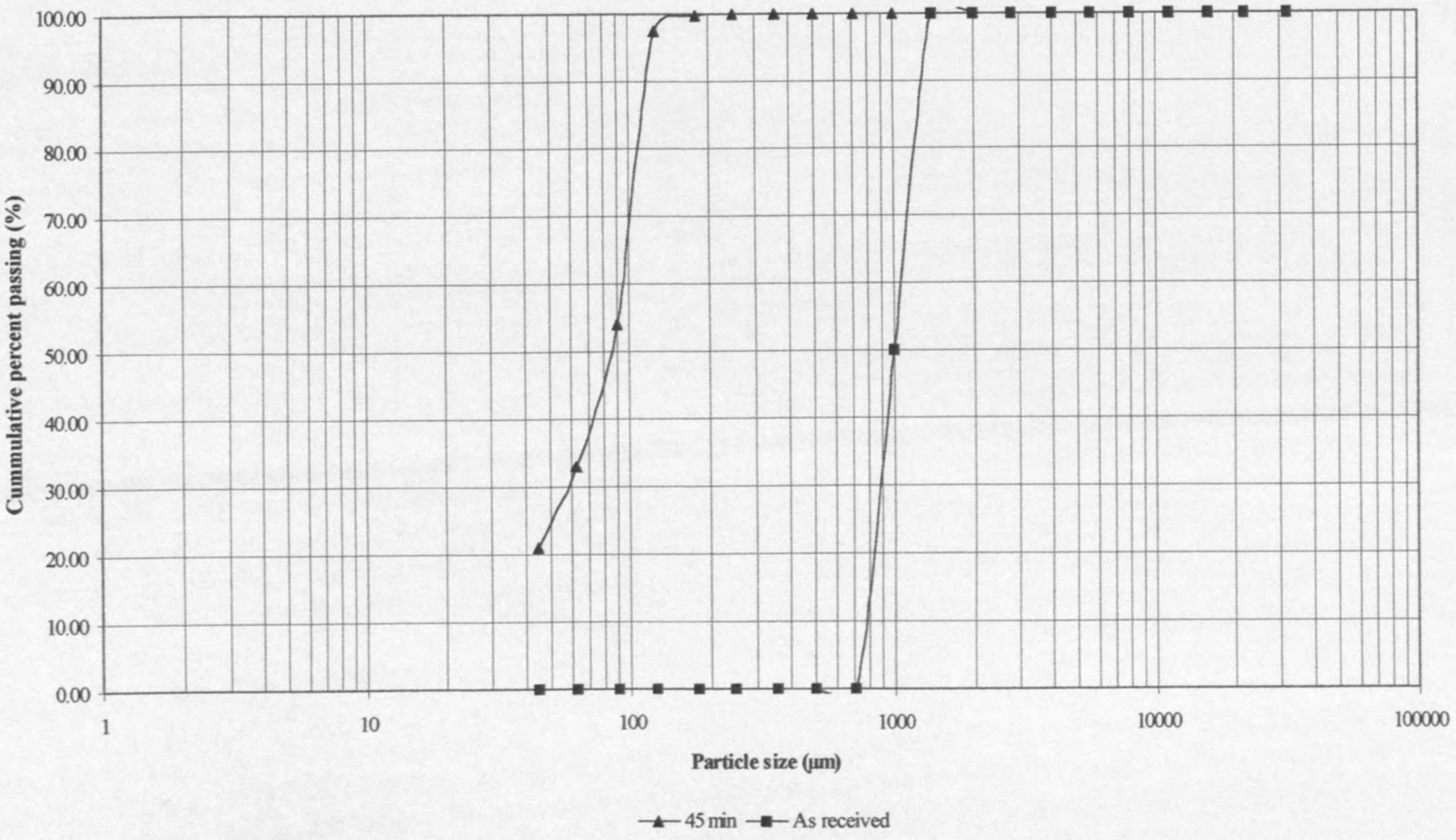
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -2.8 +2.0mm

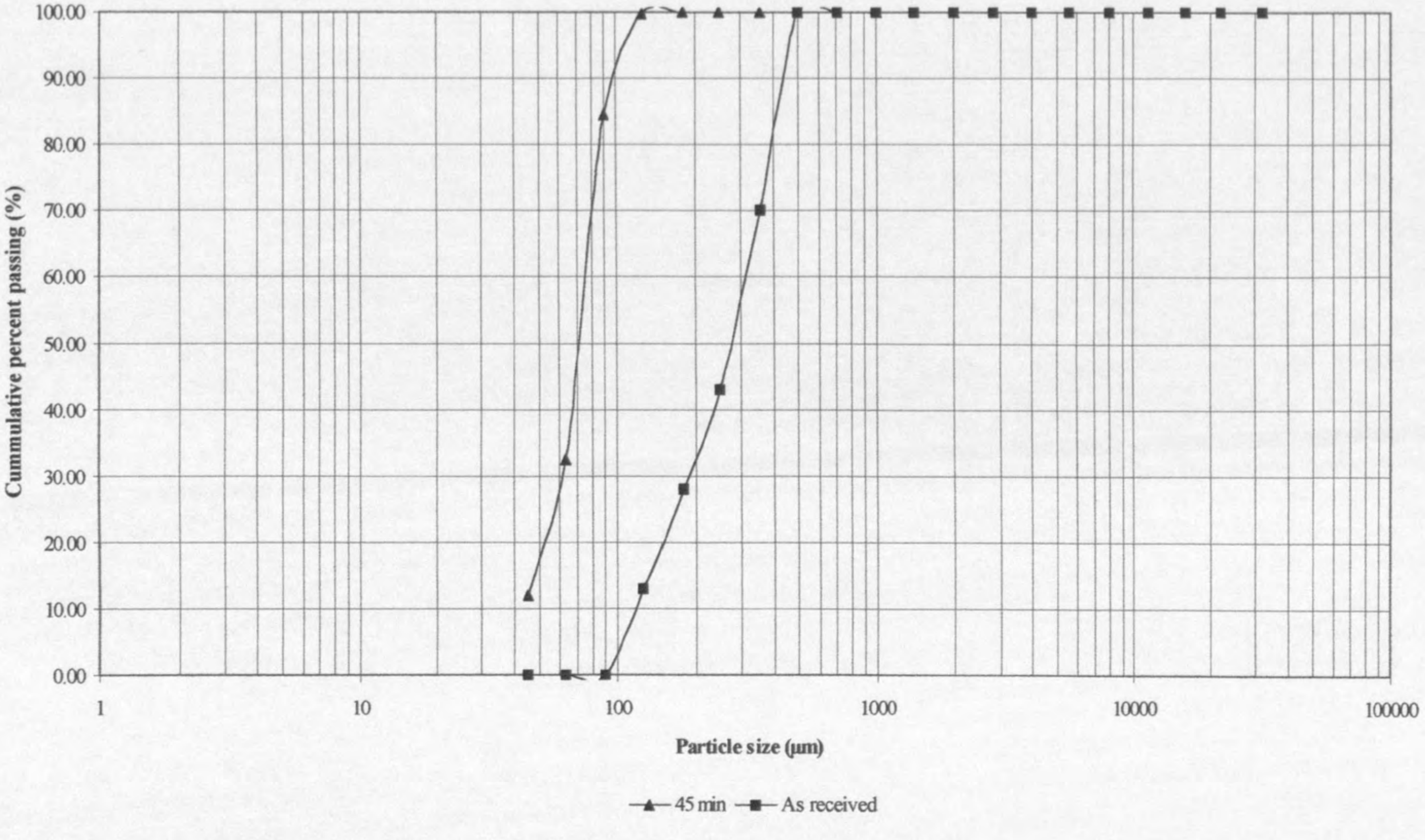




P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -1.4 +0.71mm

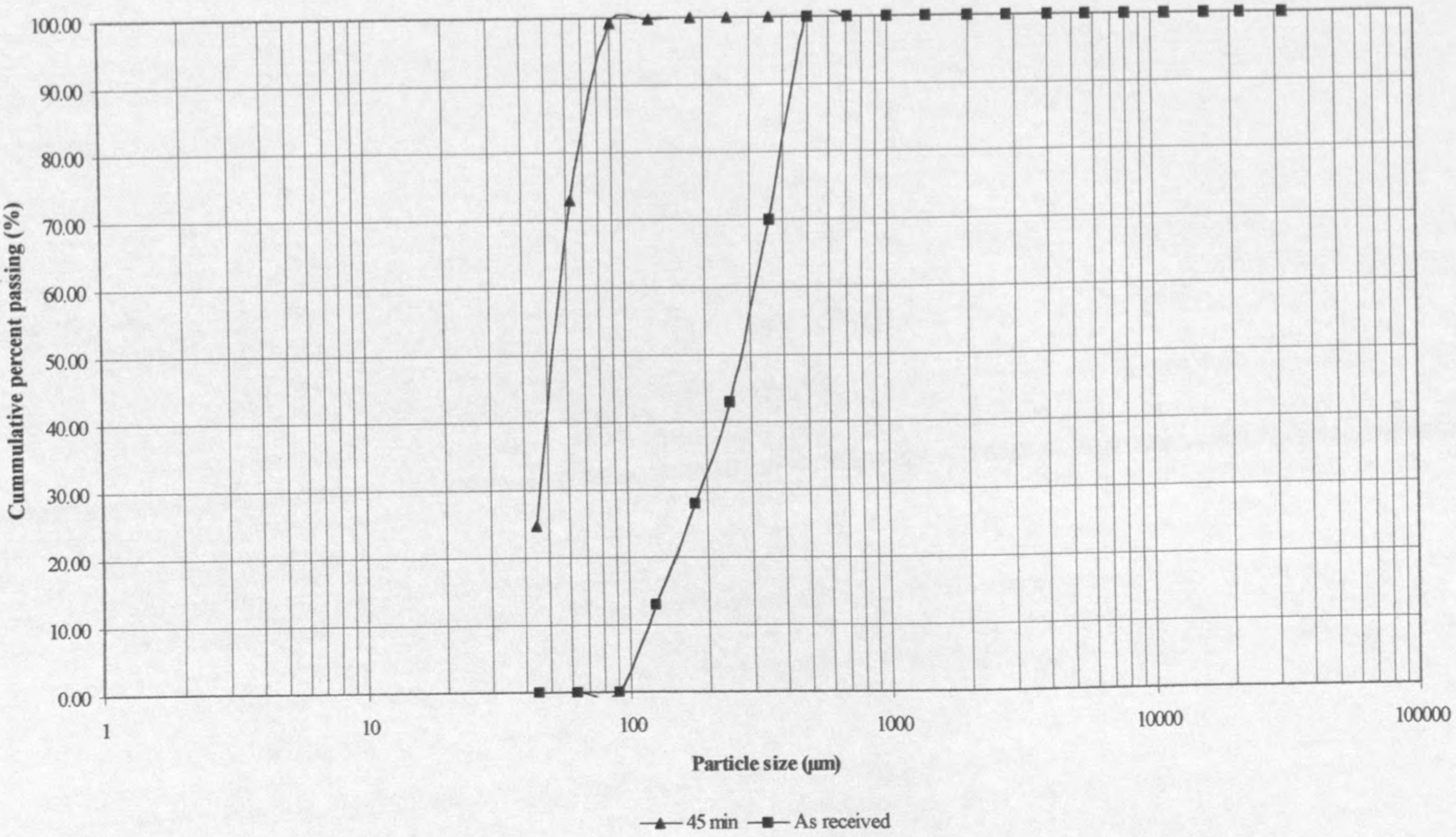
P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -1.4 +0.71mm





P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
size range -0.5 +0.09mm

P-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes
size range -0.5 +0.09mm

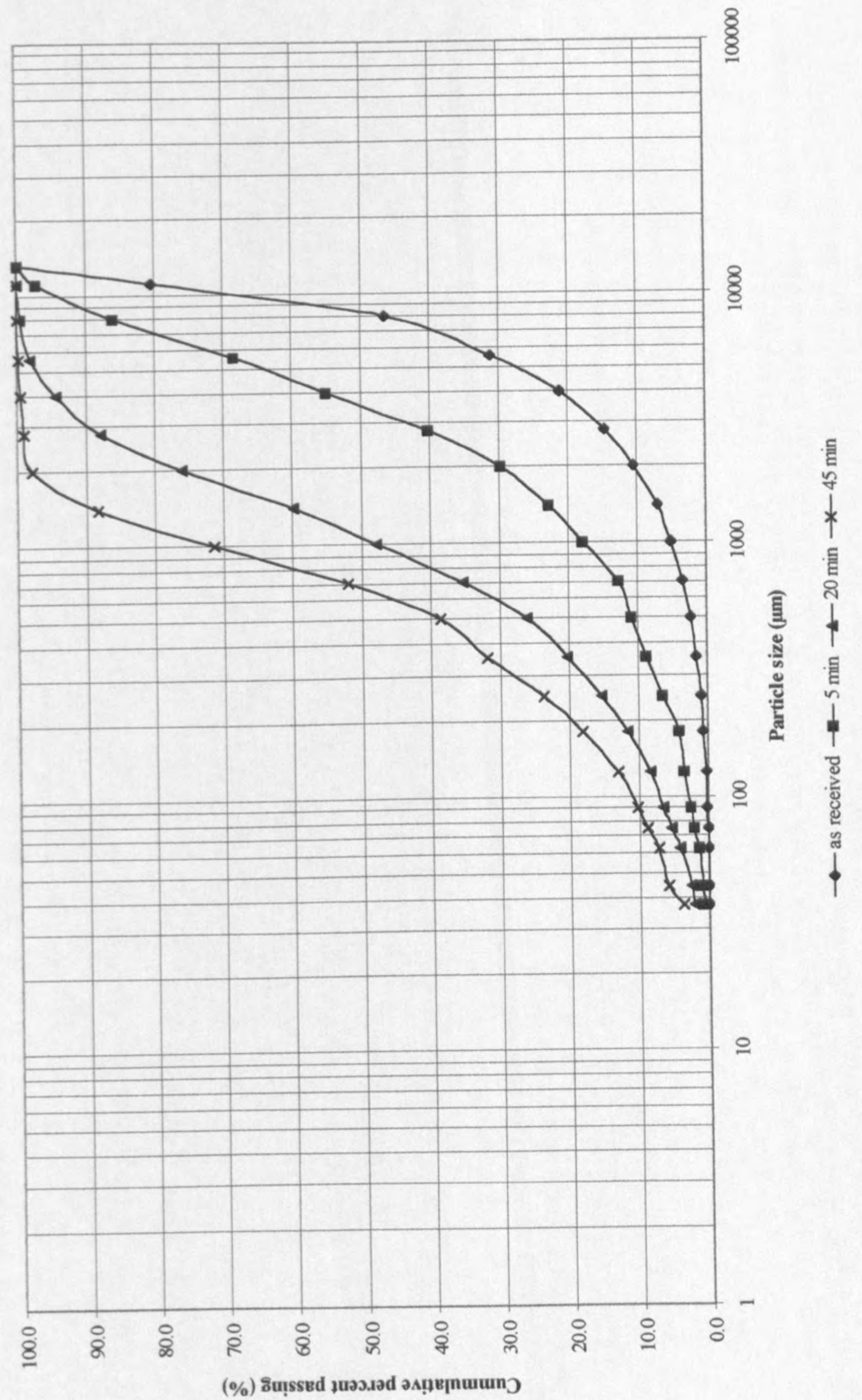


APPENDIX D8

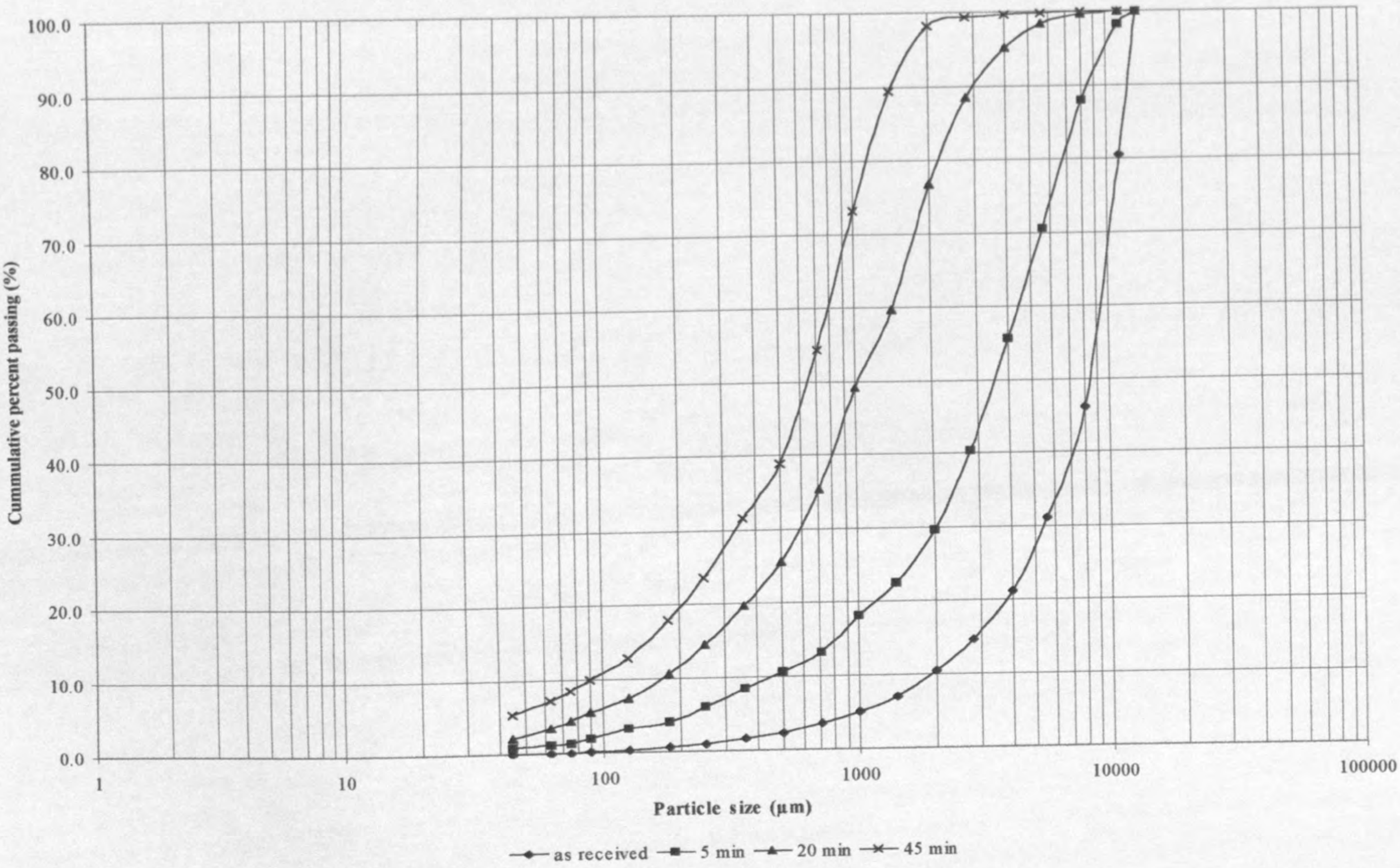
MICROWAVE TREATED GROUP 1 COALS
COAL RANK TESTS
ROD MILL SIZE DISTRIBUTIONS

F-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

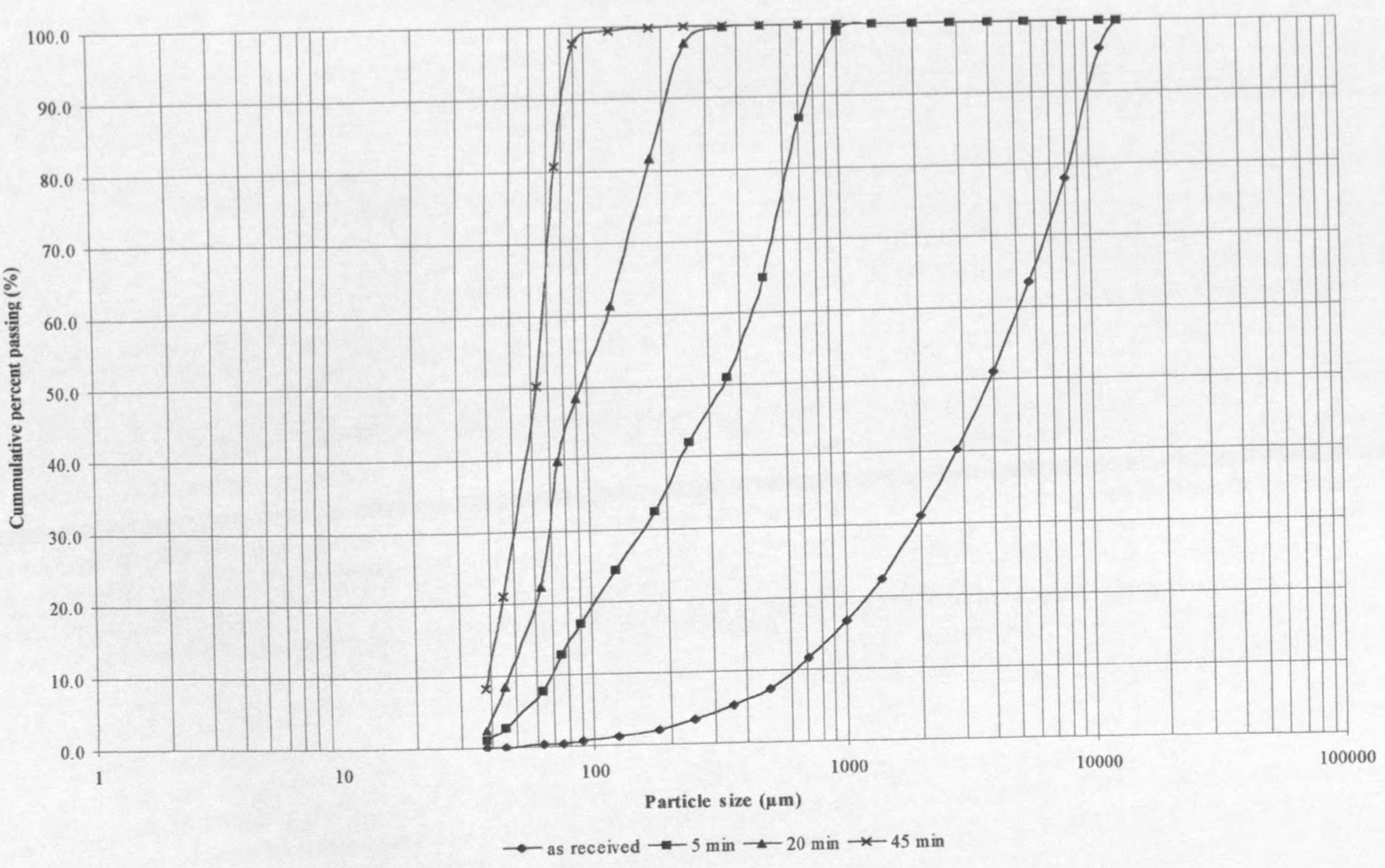
As-received



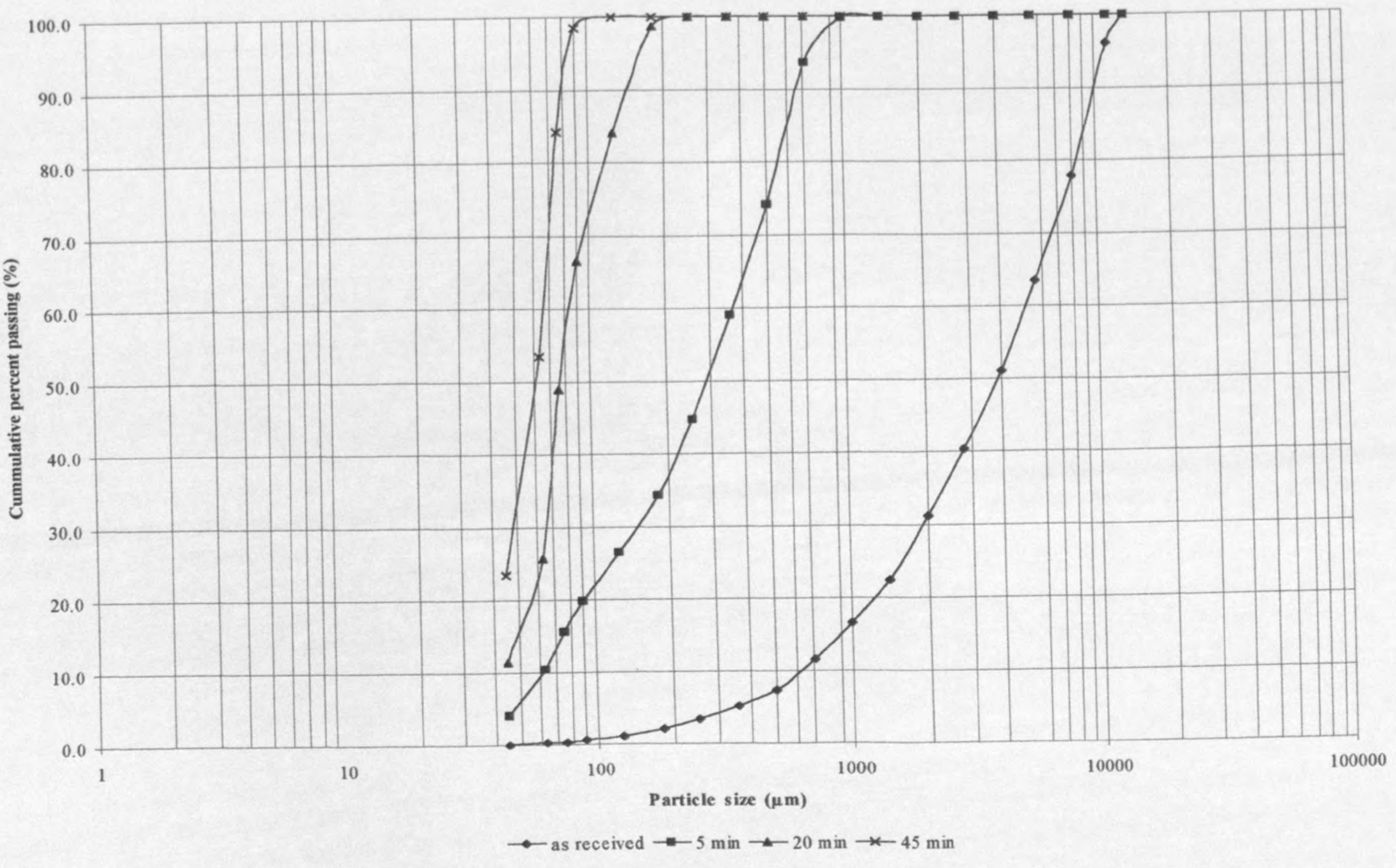
F-1 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave inpur power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



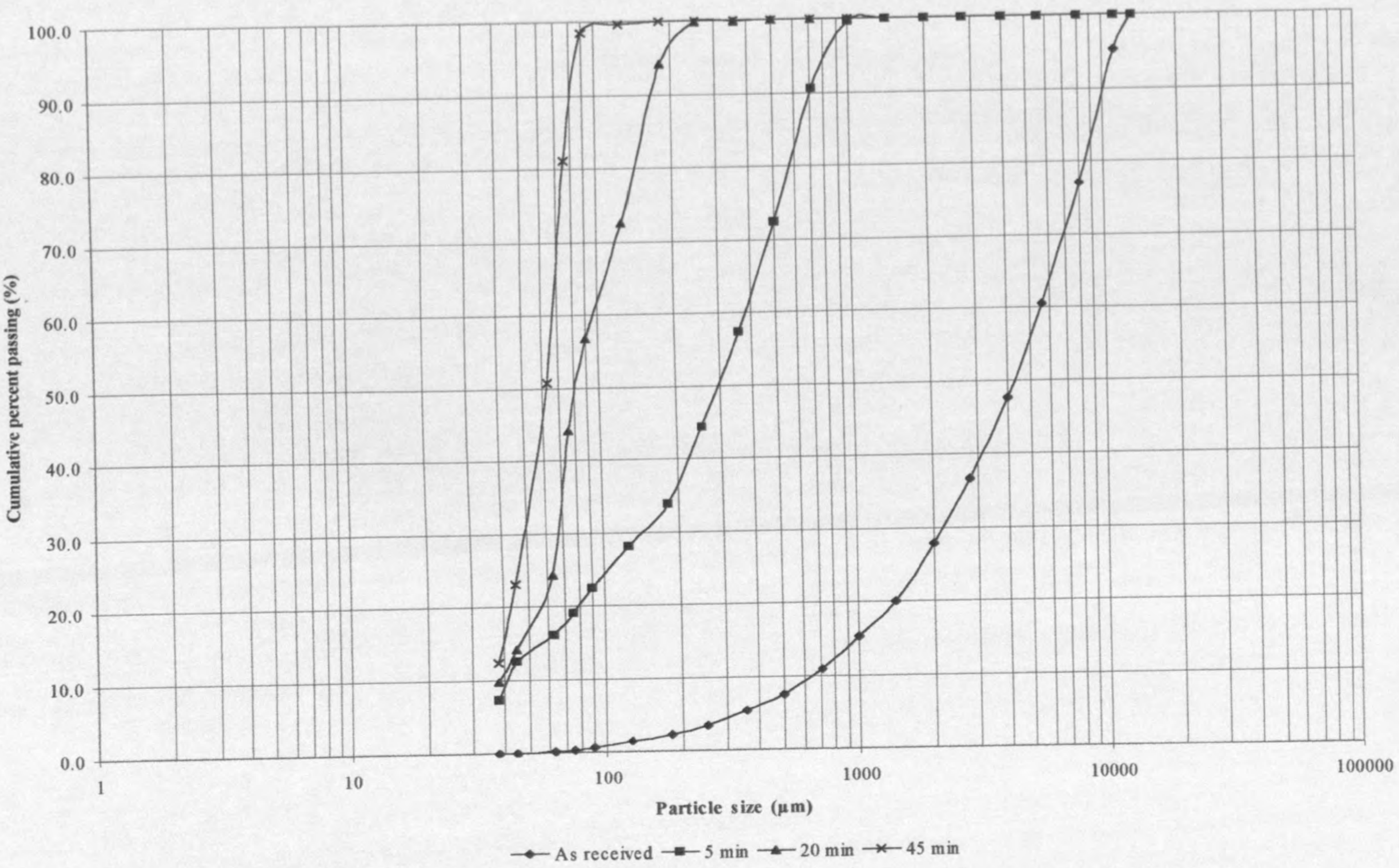
F-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



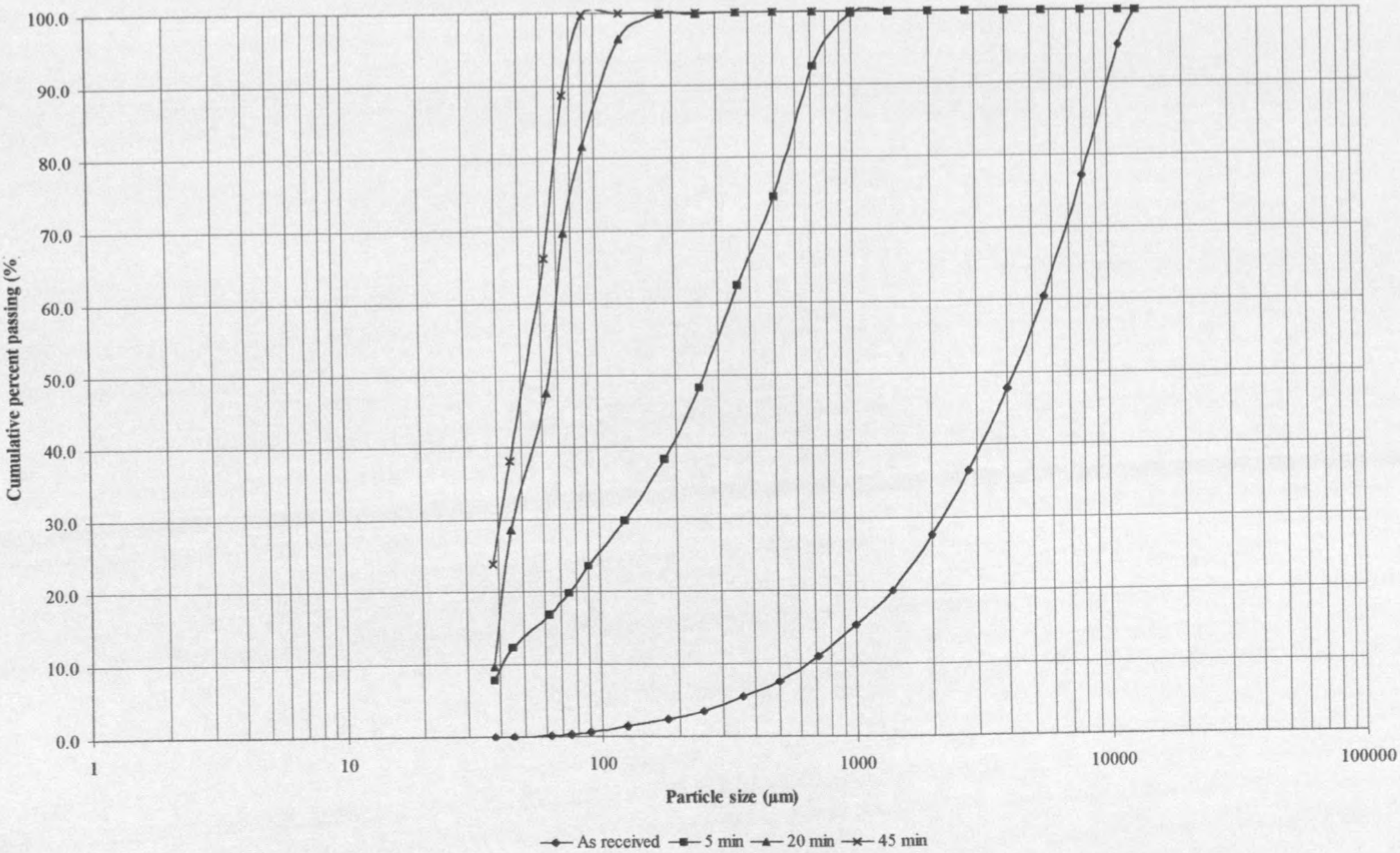
F-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, appied frequency-2.45GHz, exposure time-8 minutes



F-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

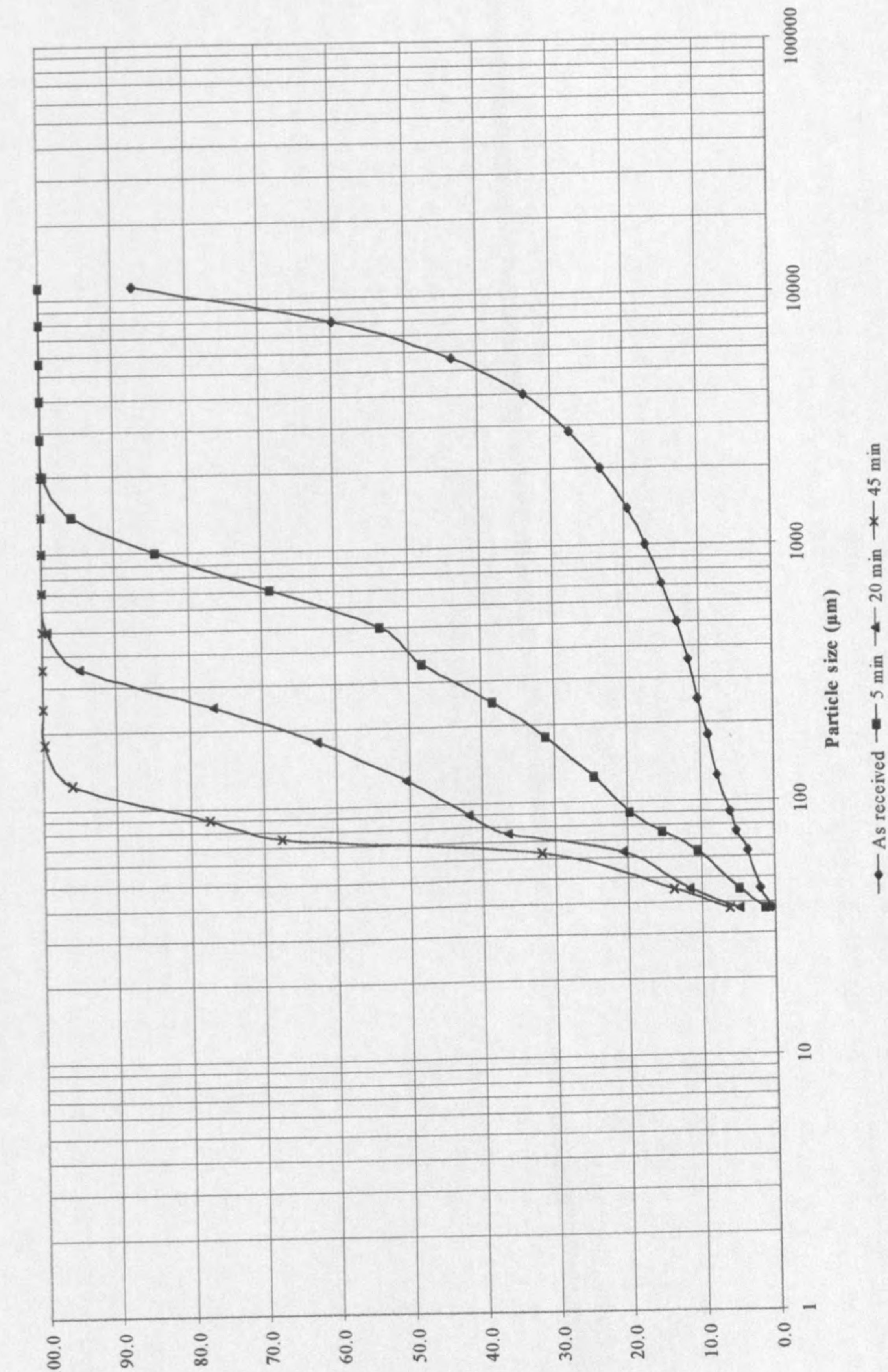


F-3 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes

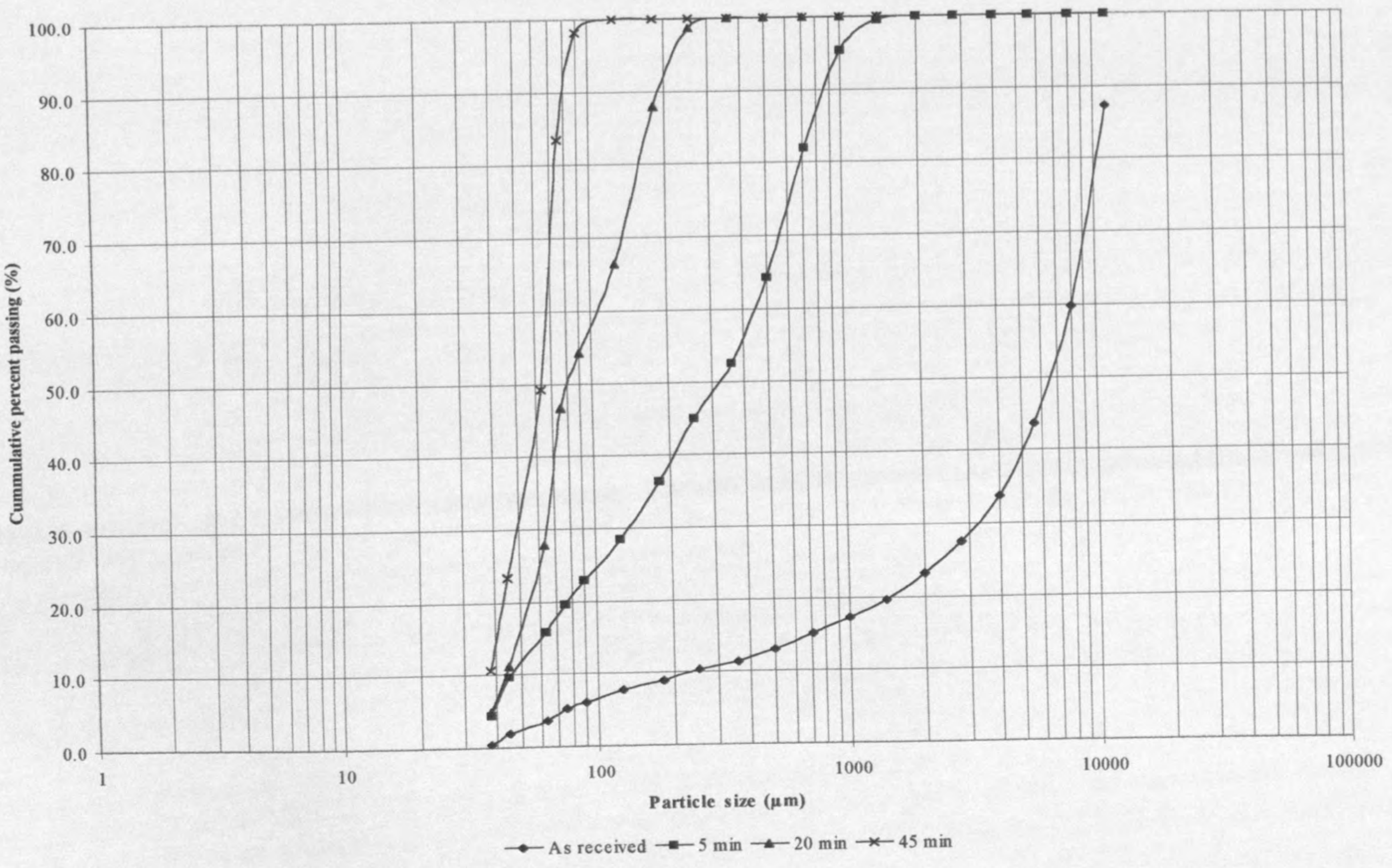


F-4 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

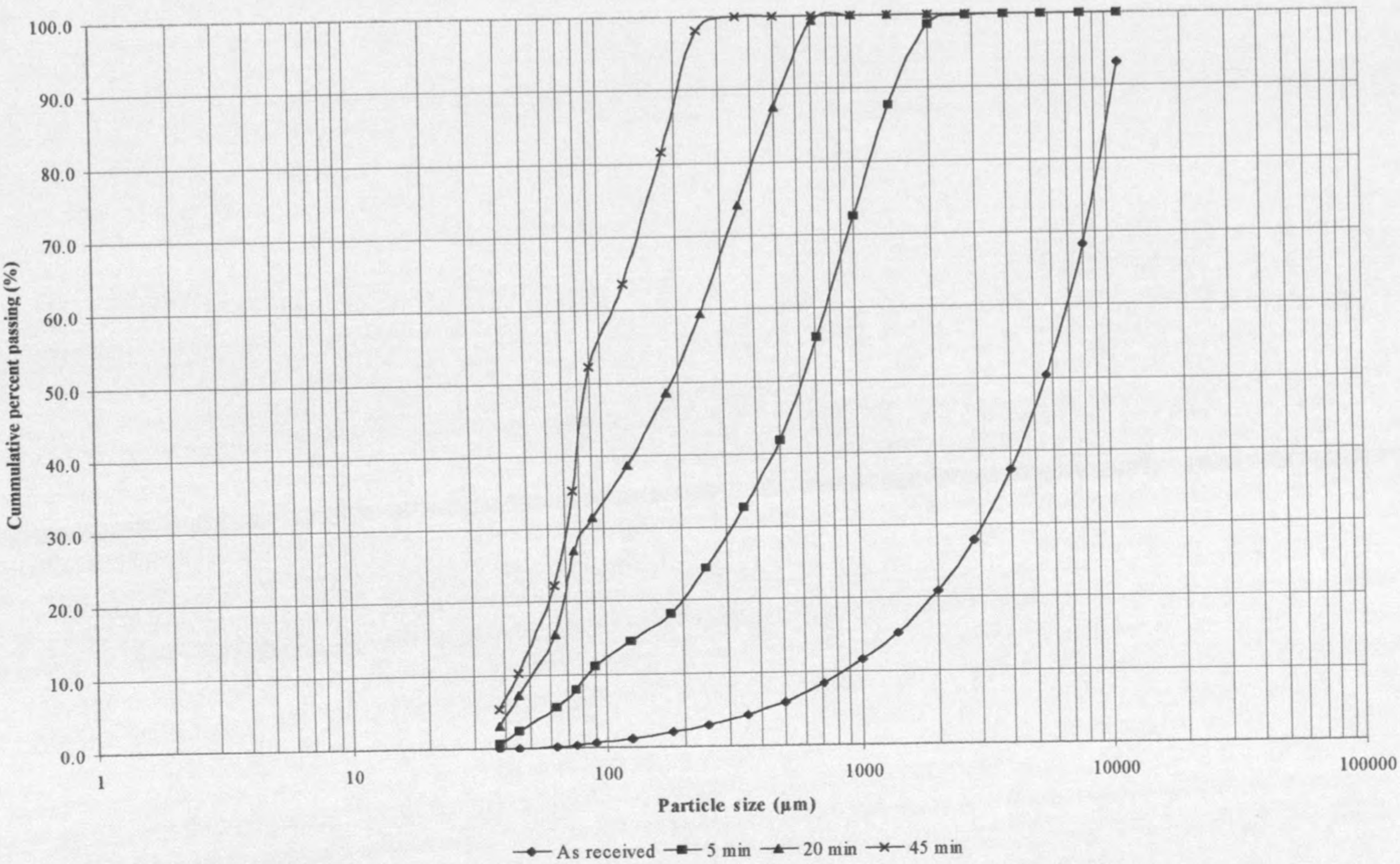
As-received

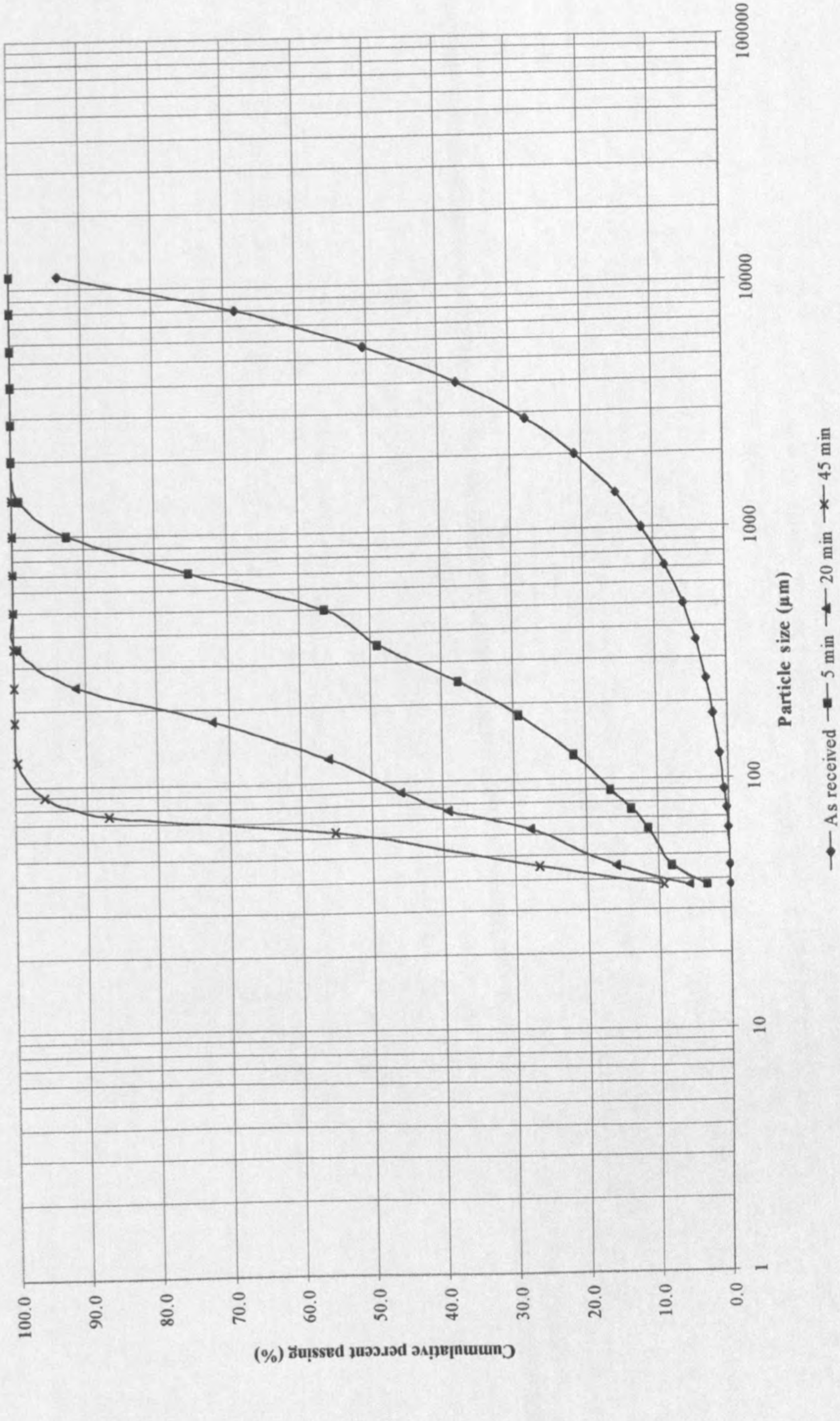


F-4 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes

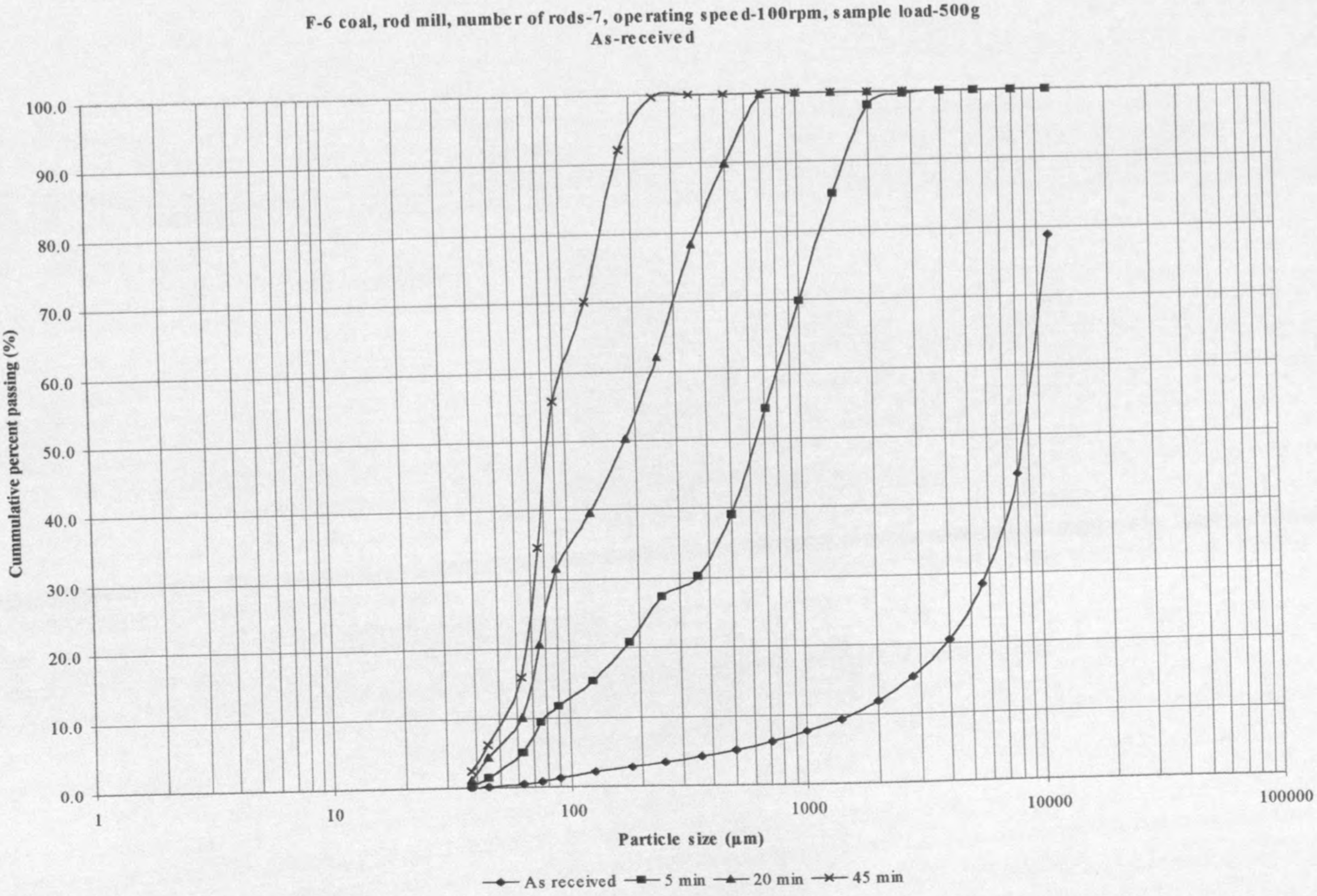


F-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

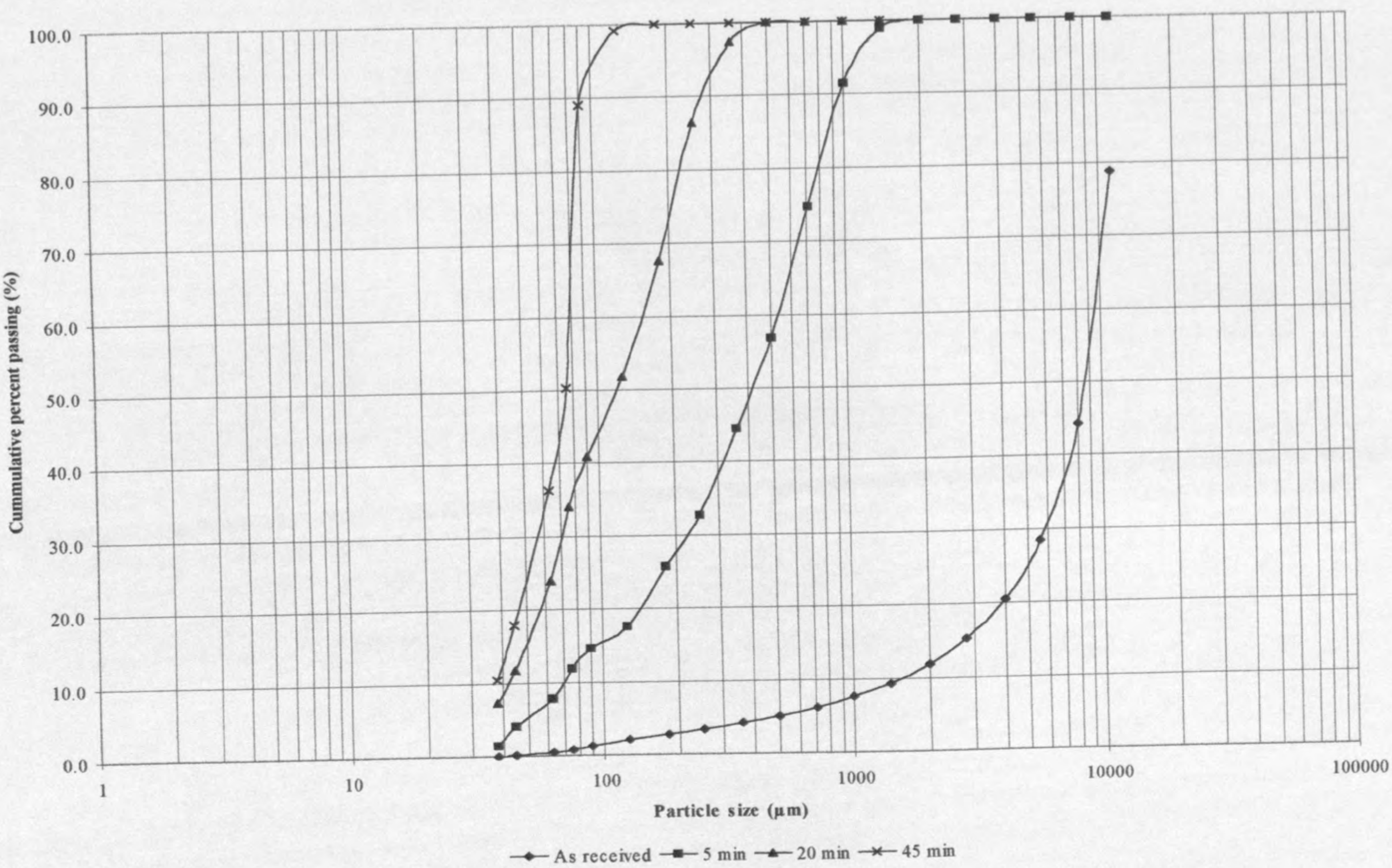




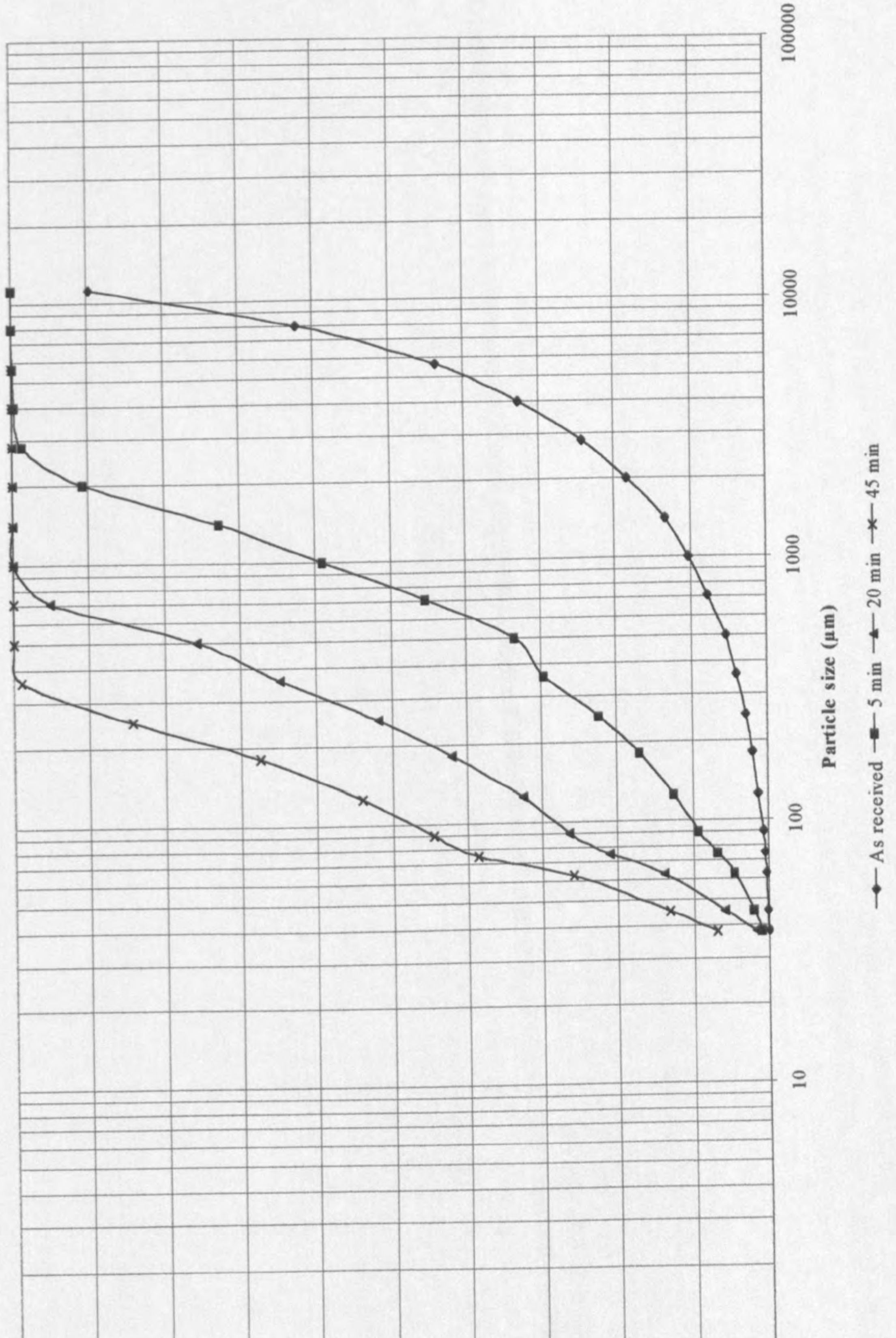
F-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes


F-6 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received
Cummulative percent passing (%)
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
1
10
100
1000
10000
100000
Particle size (µm)
As received
5 min
20 min
45 min

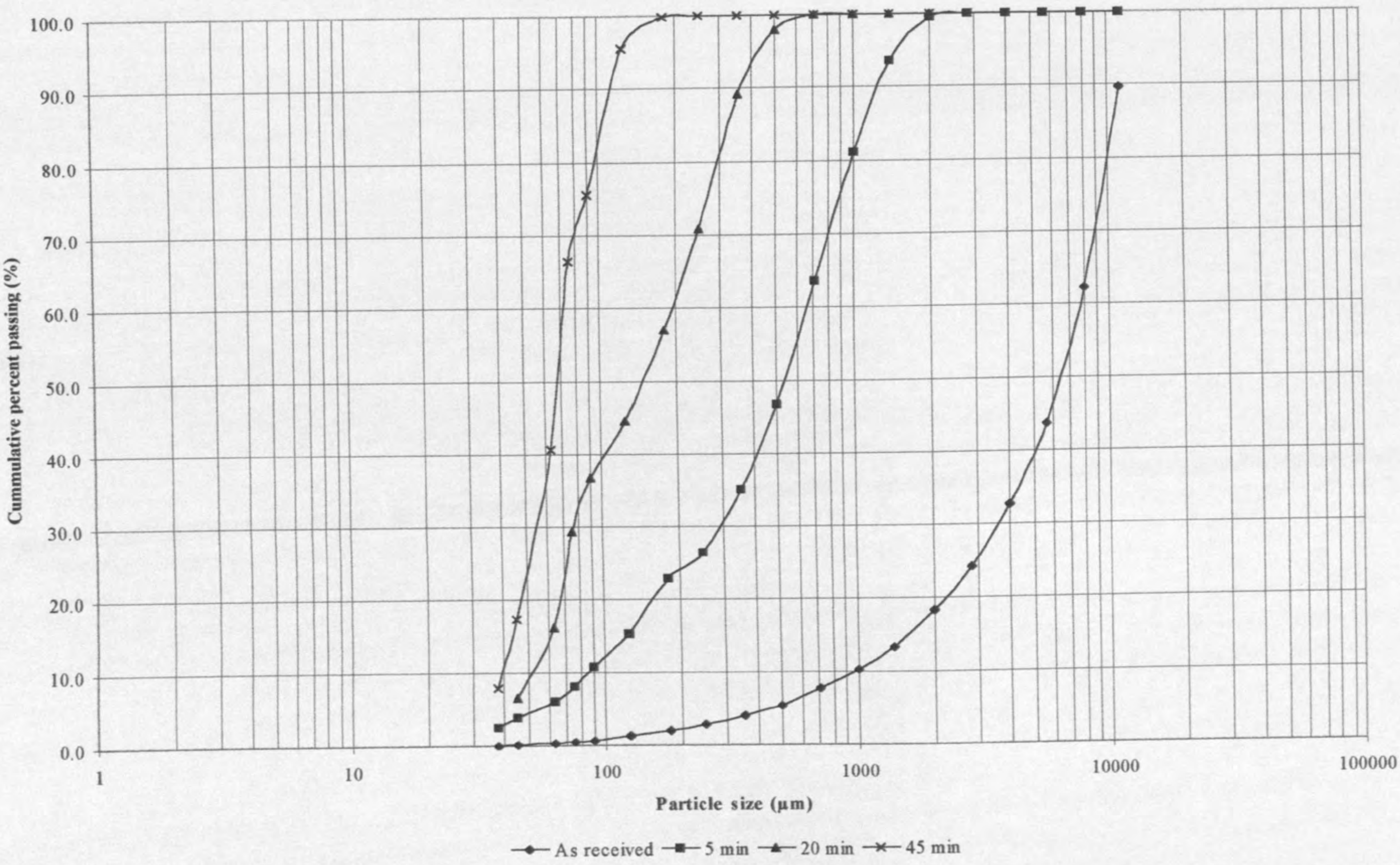
F-6 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



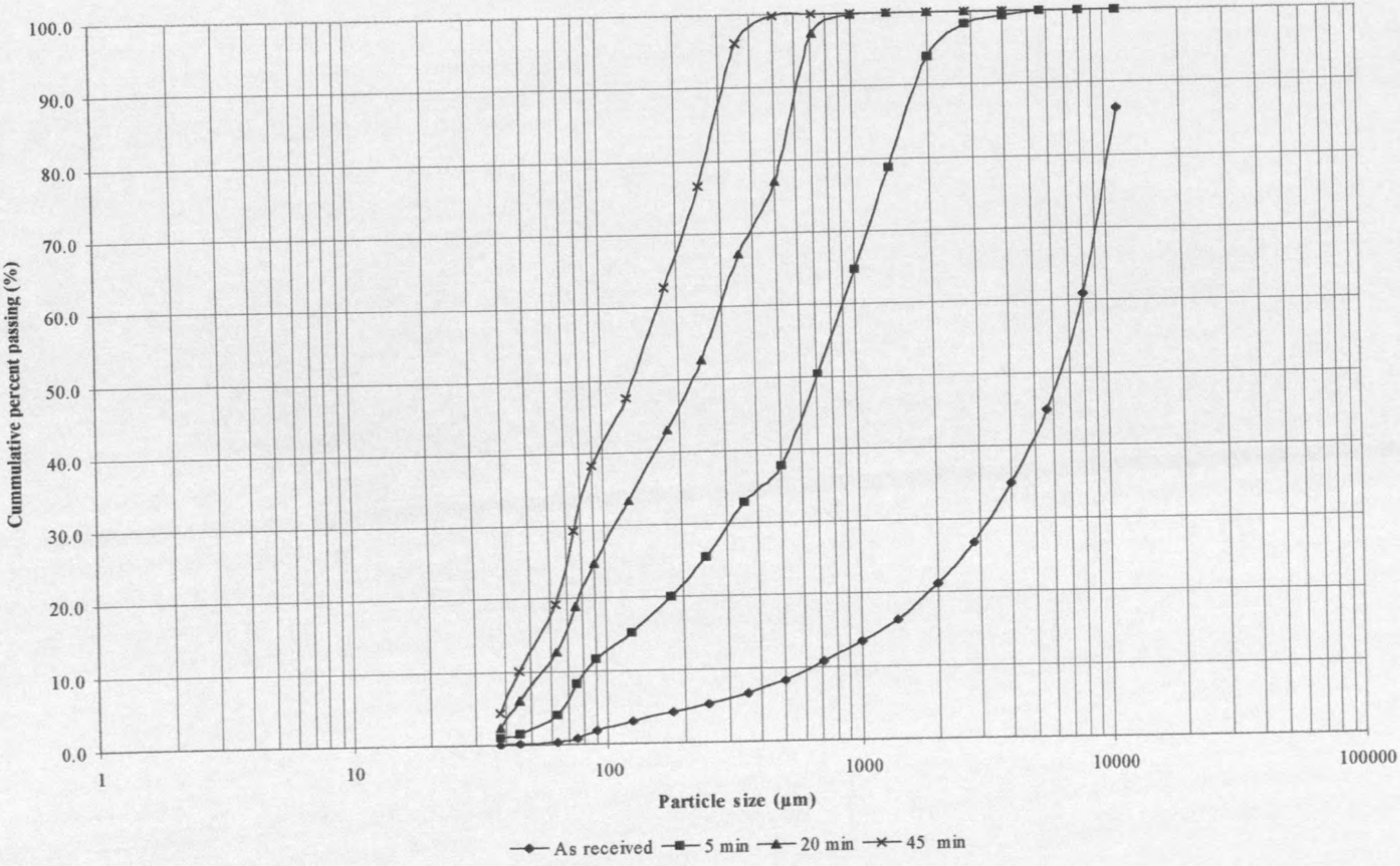
F-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



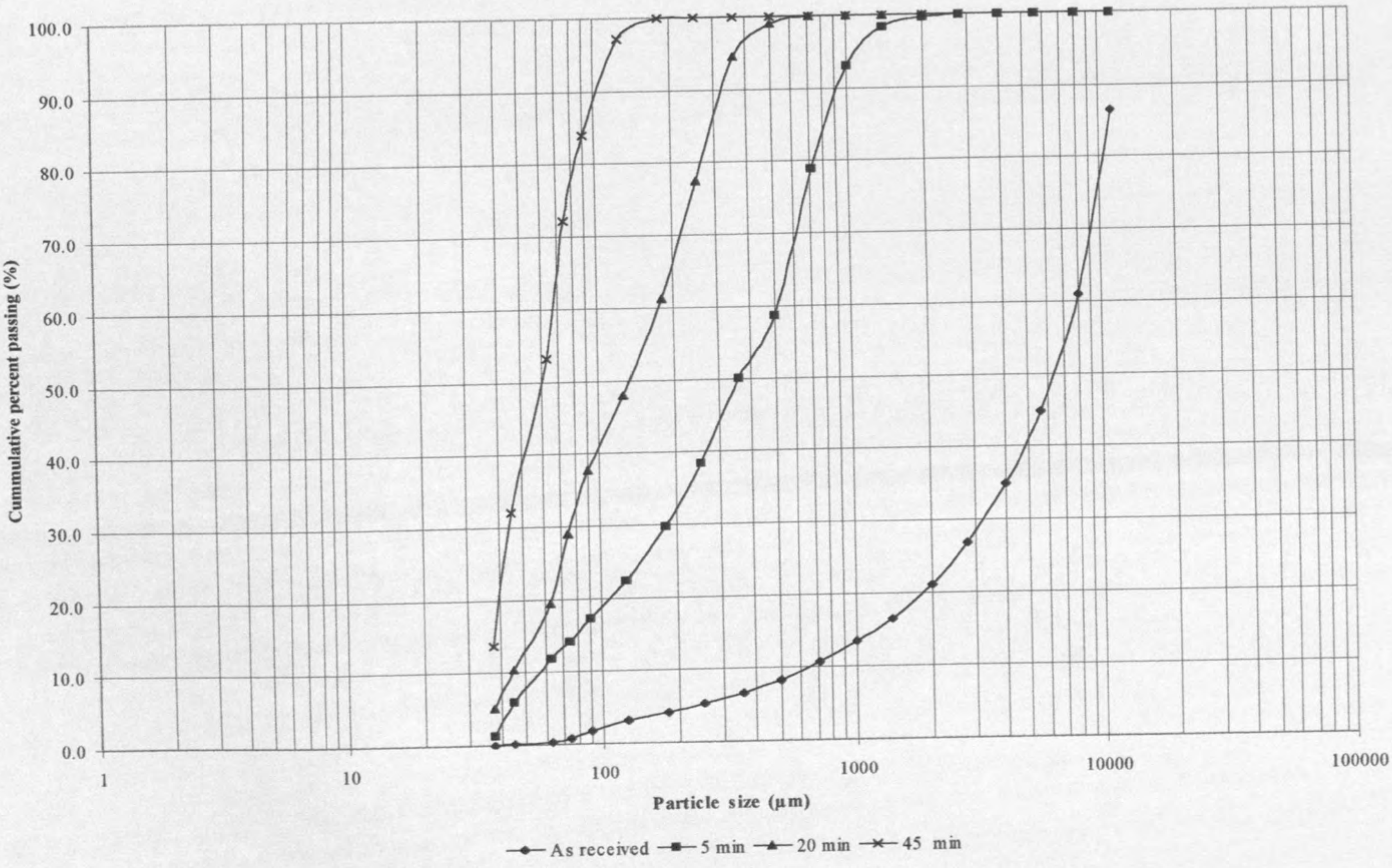
F-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



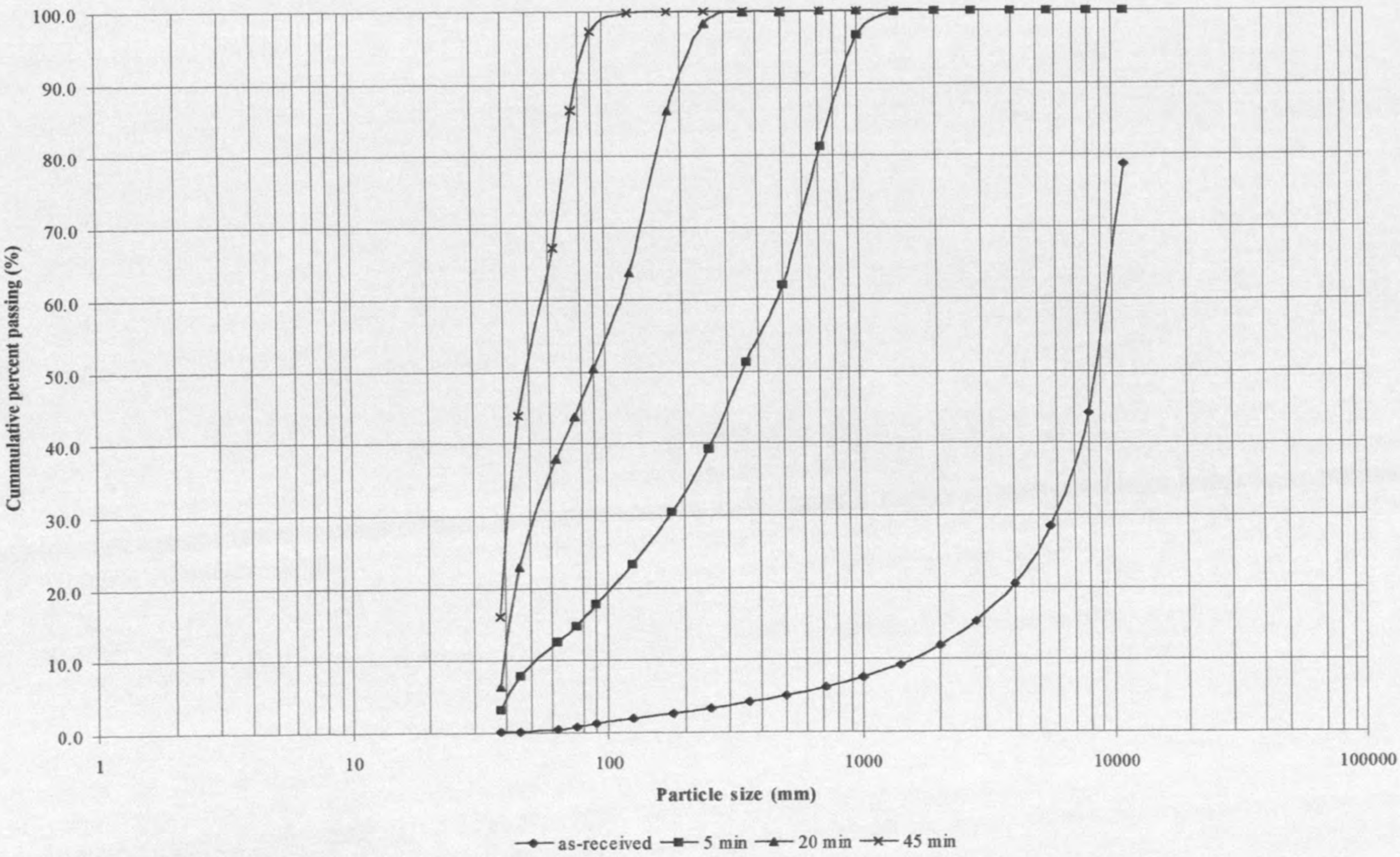
F-8 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

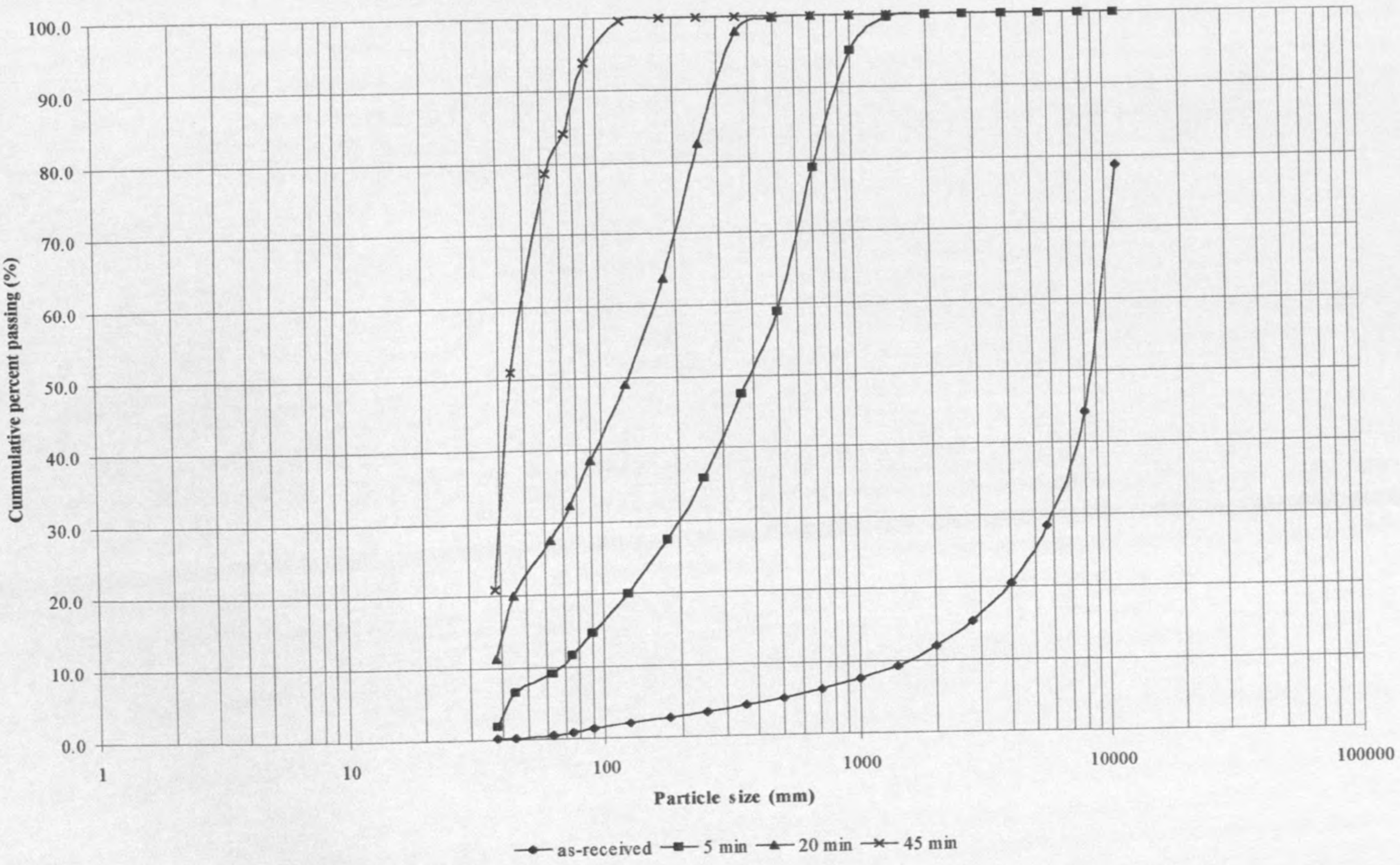


F-8 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes

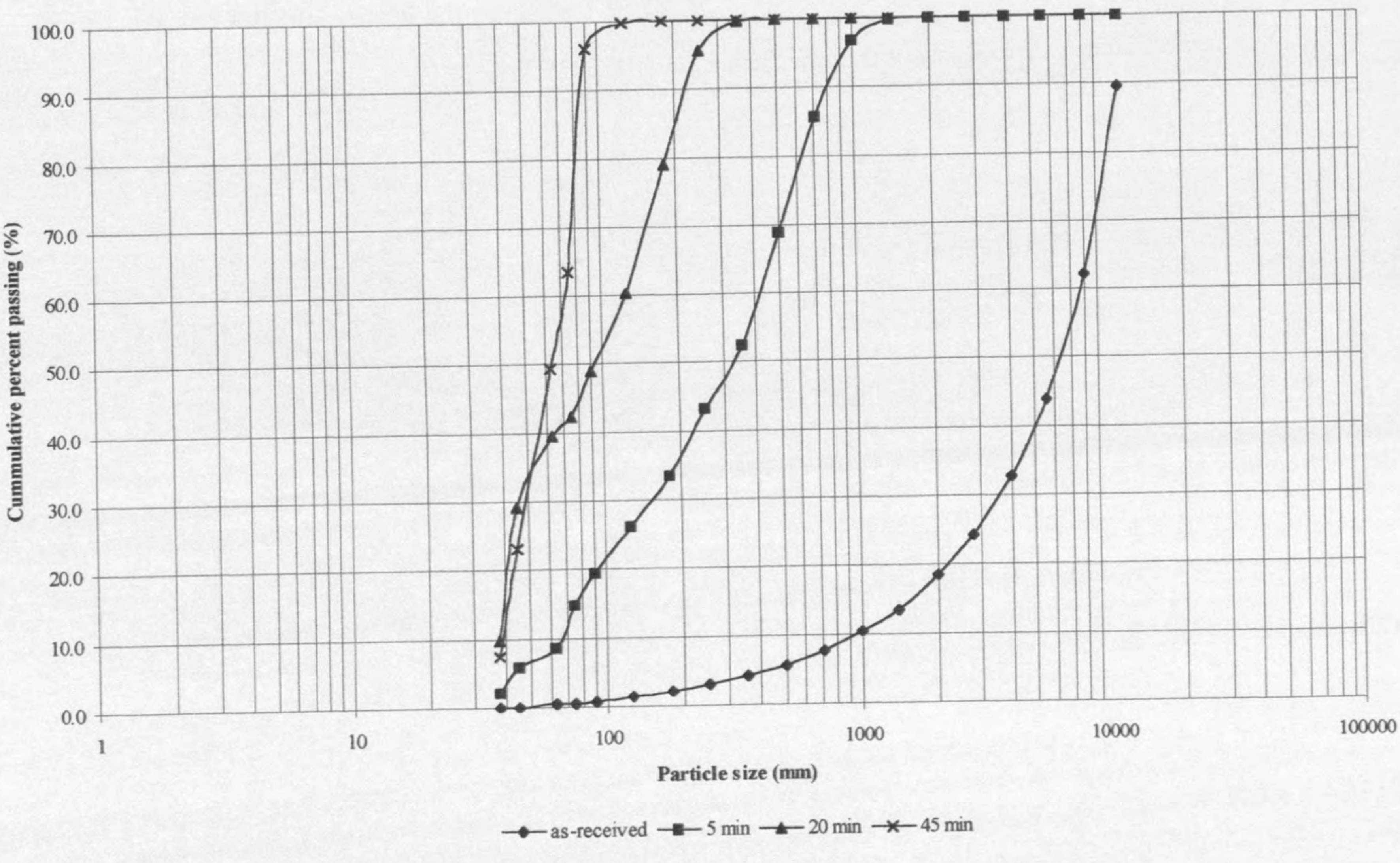


P-6 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-100°C, exposure time-1 hour



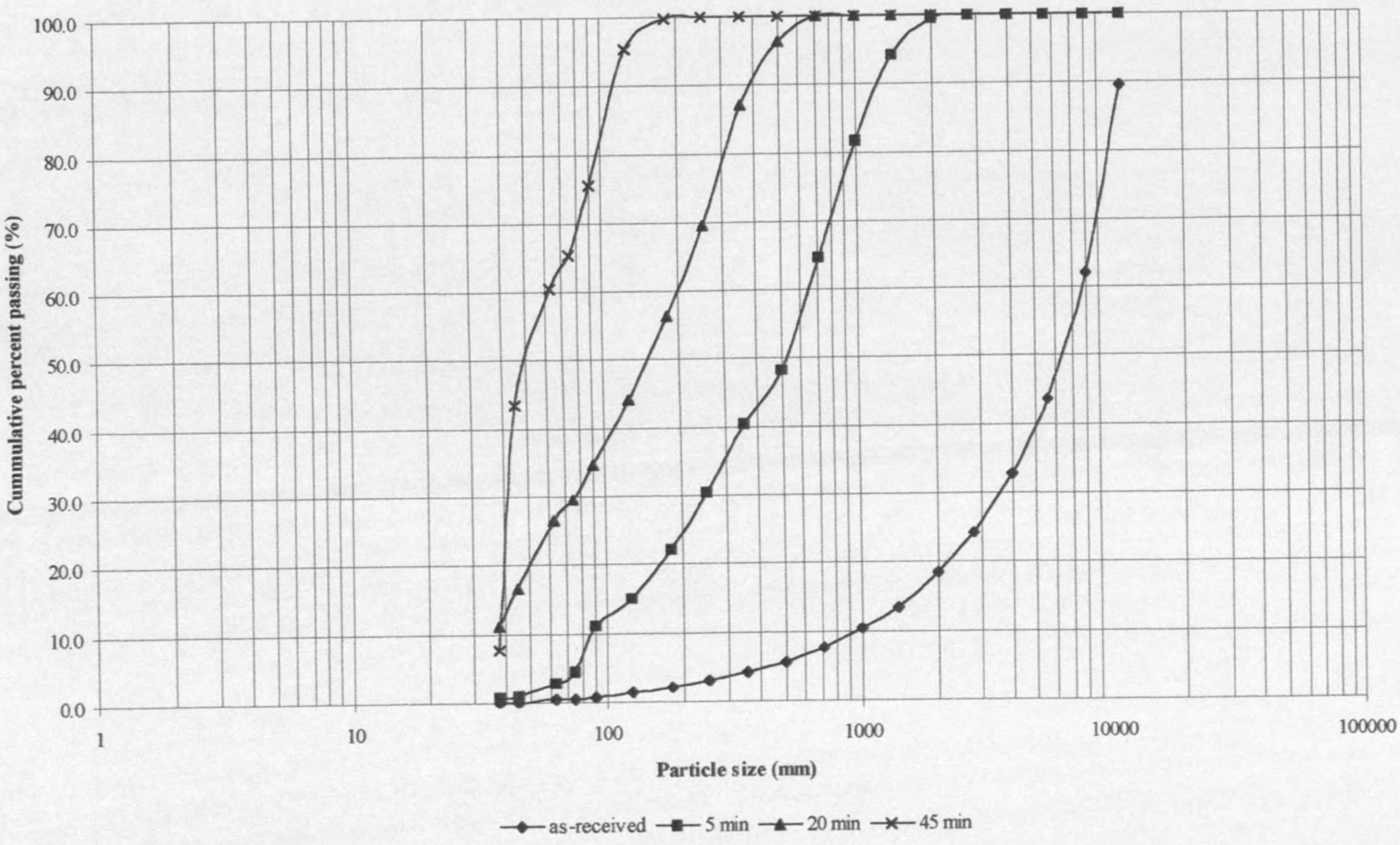

F-6 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-200°C, exposure time-1 hour

F-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-100°C, exposure time-1 hour



F-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

Muffle furnace temperature-200°C, exposure time-1 hour



APPENDIX D9

MICROWAVE TREATED COALS AT CONSTANT ENERGY INPUT MOISTURE ADDITION TESTS ROD MILL SIZE DISTRIBUTIONS

P-7 coal, rod mill, number of rods-7, operating speed-100rpm, load-500g, D9-13
Muffle furnace temperature-110°C, exposure time-3 hours
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

P-7 coal sample A, rod mill, number of rods-7, operating speed-100rpm, D9-14
load-500g, addition of 400ml water 50g Calgon solution
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

P-7 coal sample B, rod mill, number of rods-7, operating speed-100rpm, D9-15
load-500g, addition of 400ml water 50g Calgon solution
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

P-2 coal, rod mill, number of rods-7, operating speed-100rpm, load-500g, D9-16
As-received coal

P-2 coal, rod mill, number of rods-7, operating speed-100rpm, load-500g, D9-17
Muffle furnace temperature-110°C, exposure time-3 hours

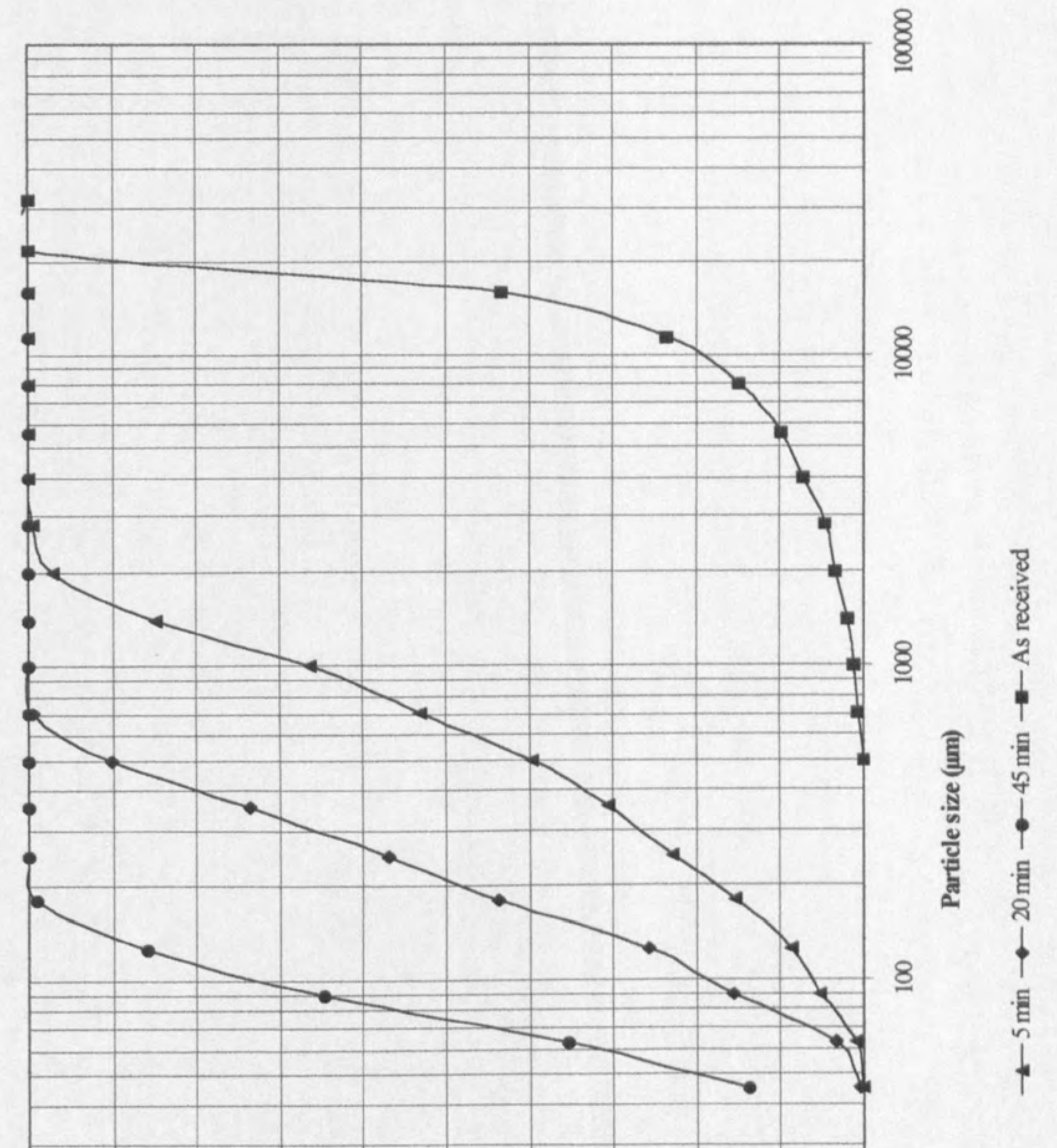
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, load-500g, D9-18
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

P-2 coal sample A, rod mill, number of rods-7, operating speed-100rpm, D9-19
load-500g, addition of 400ml water
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

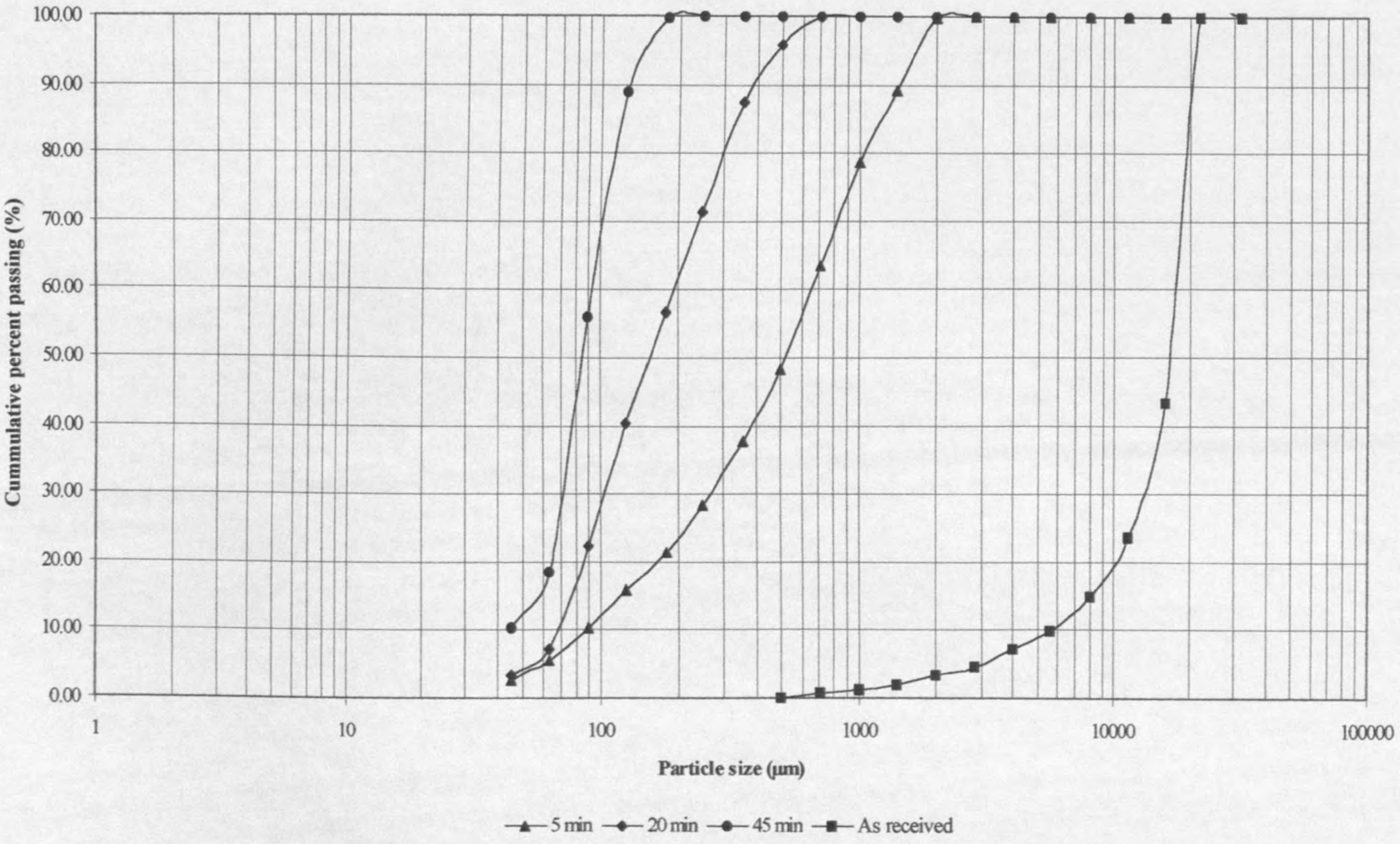
P-2 coal sample B, rod mill, number of rods-7, operating speed-100rpm, D9-20
load-500g, addition of 400ml water
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

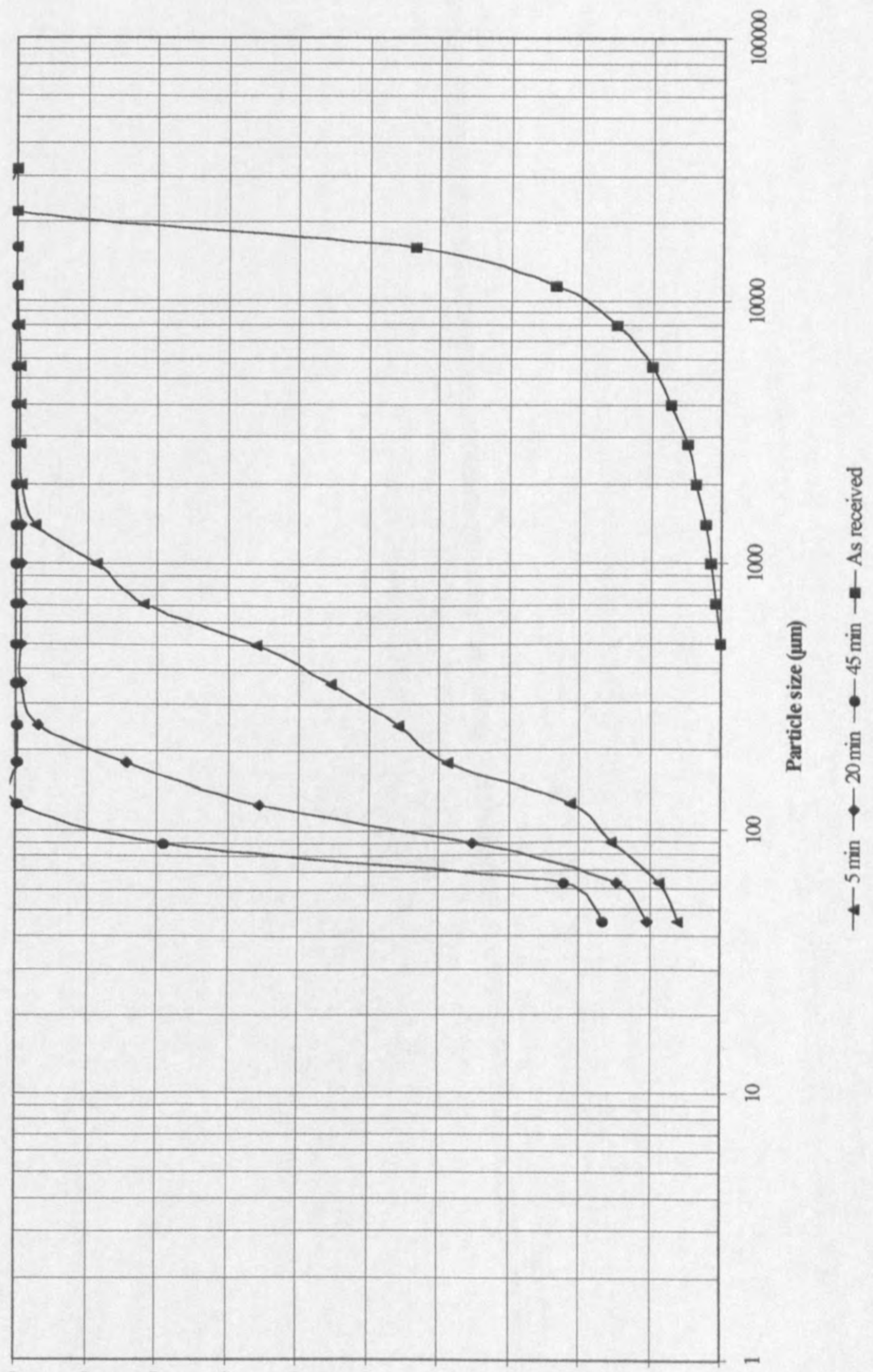
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, load-500g, D9-21
Muffle furnace temperature-110°C, exposure time-3 hours
Microwave input power-0.65kW, frequency-2.45GHz, exposure time-8 minutes

P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



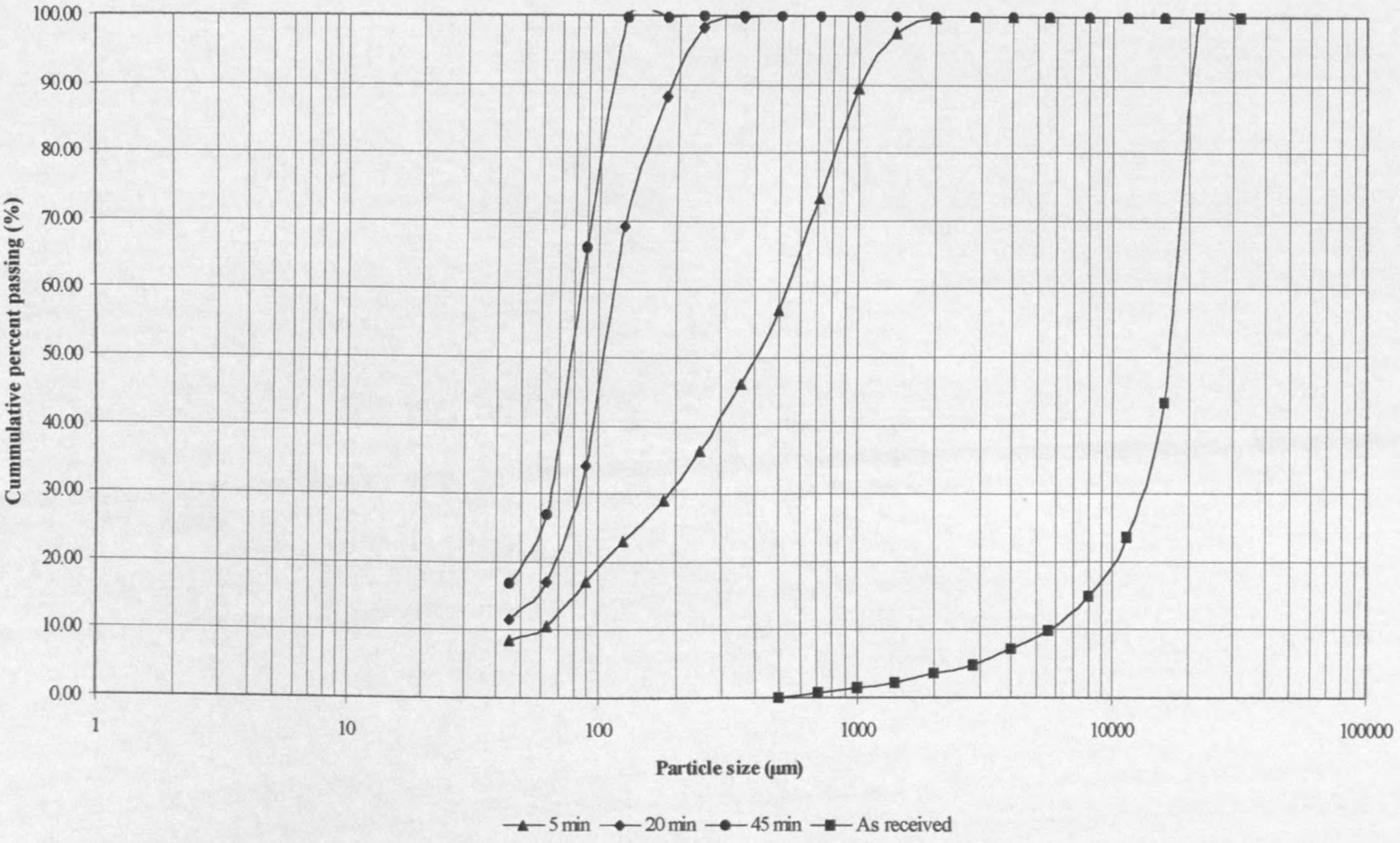
P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Covered in Ice for 1hr



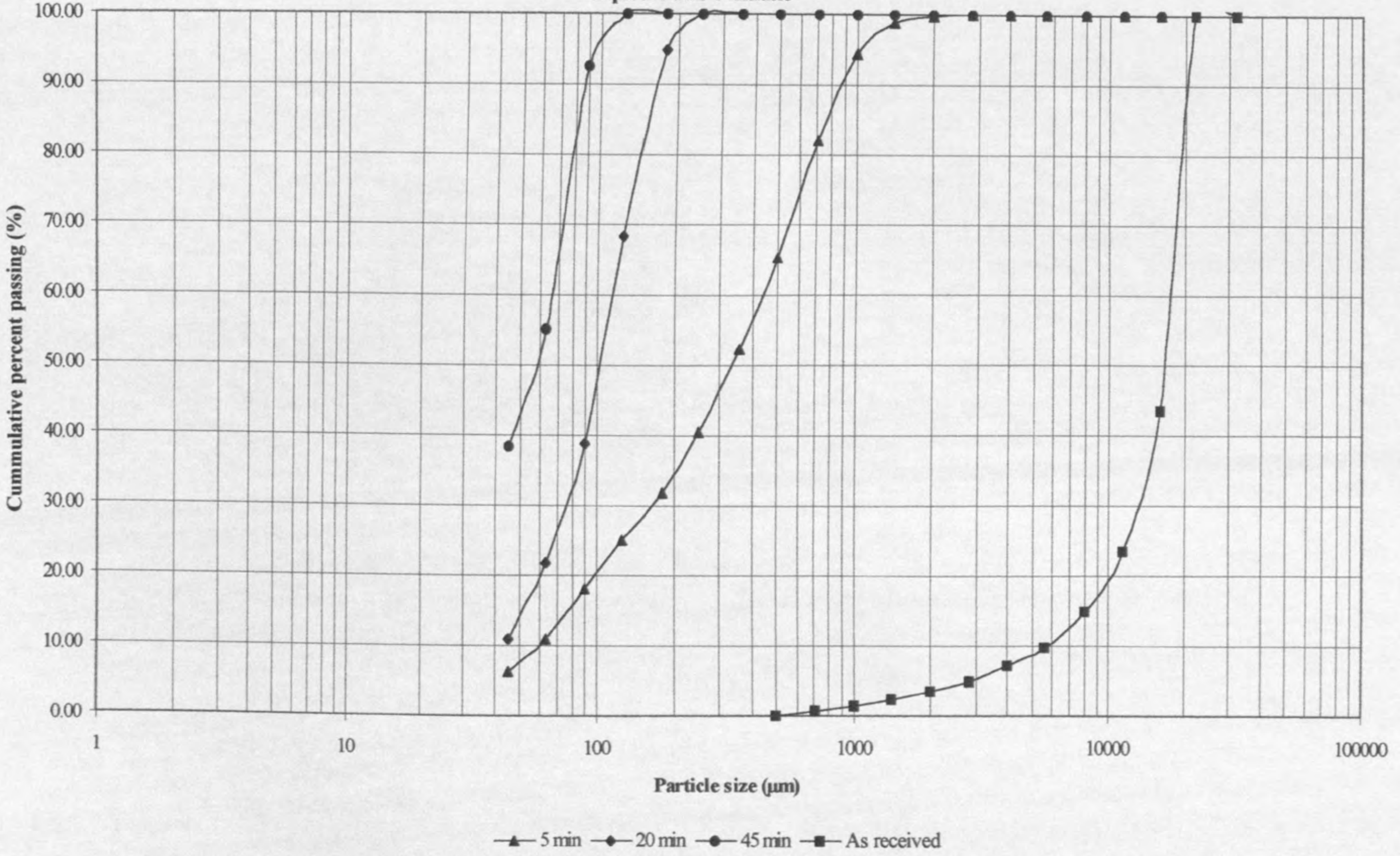


P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of liquid N_2 for 1hr

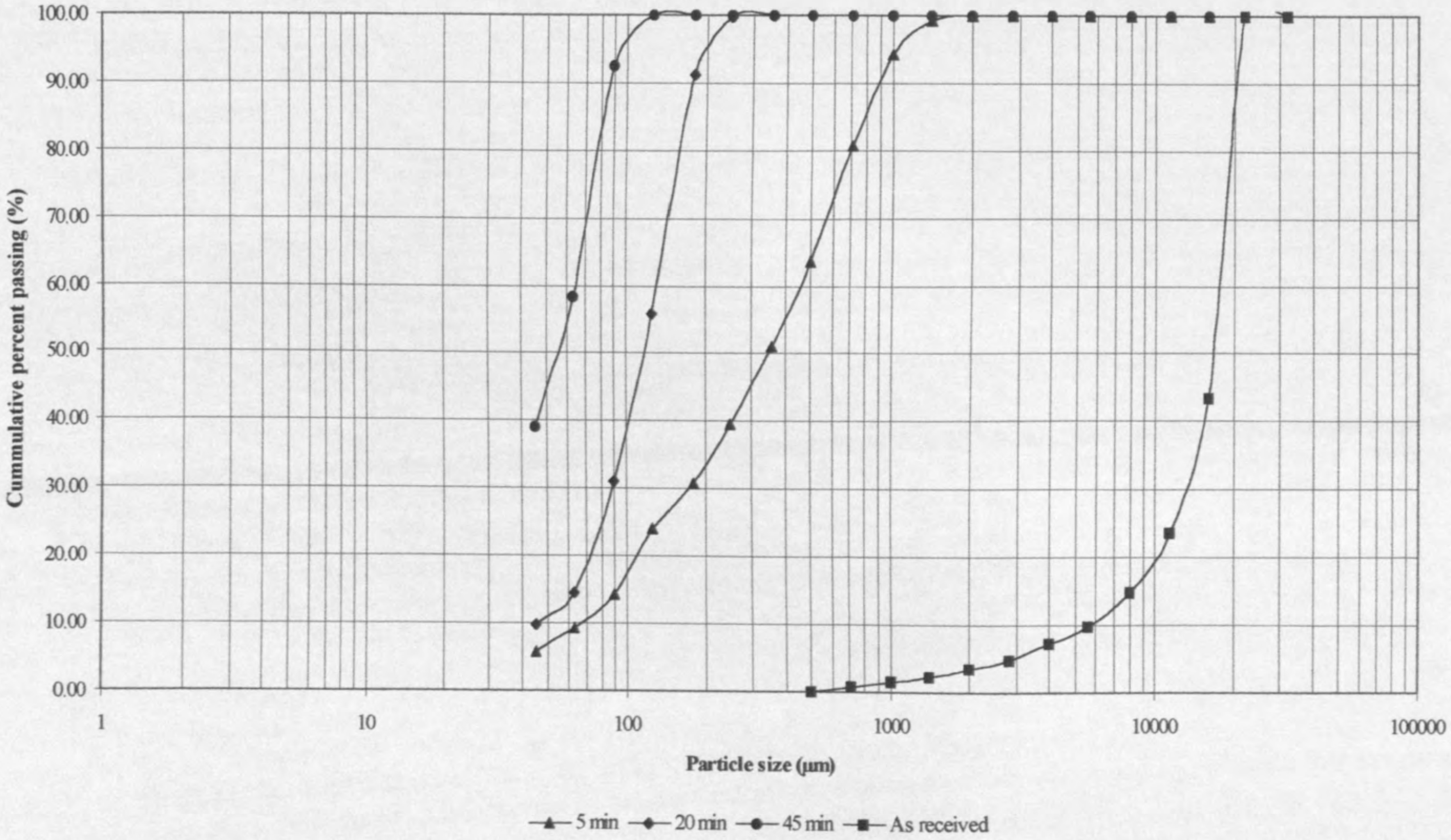
P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Steam addition for 1hr



P-7 coal sample A, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Steam addition for 1hr, Microwave input power-0.65kW, applied frequency-2.45GHz,
exposure time-8 minutes

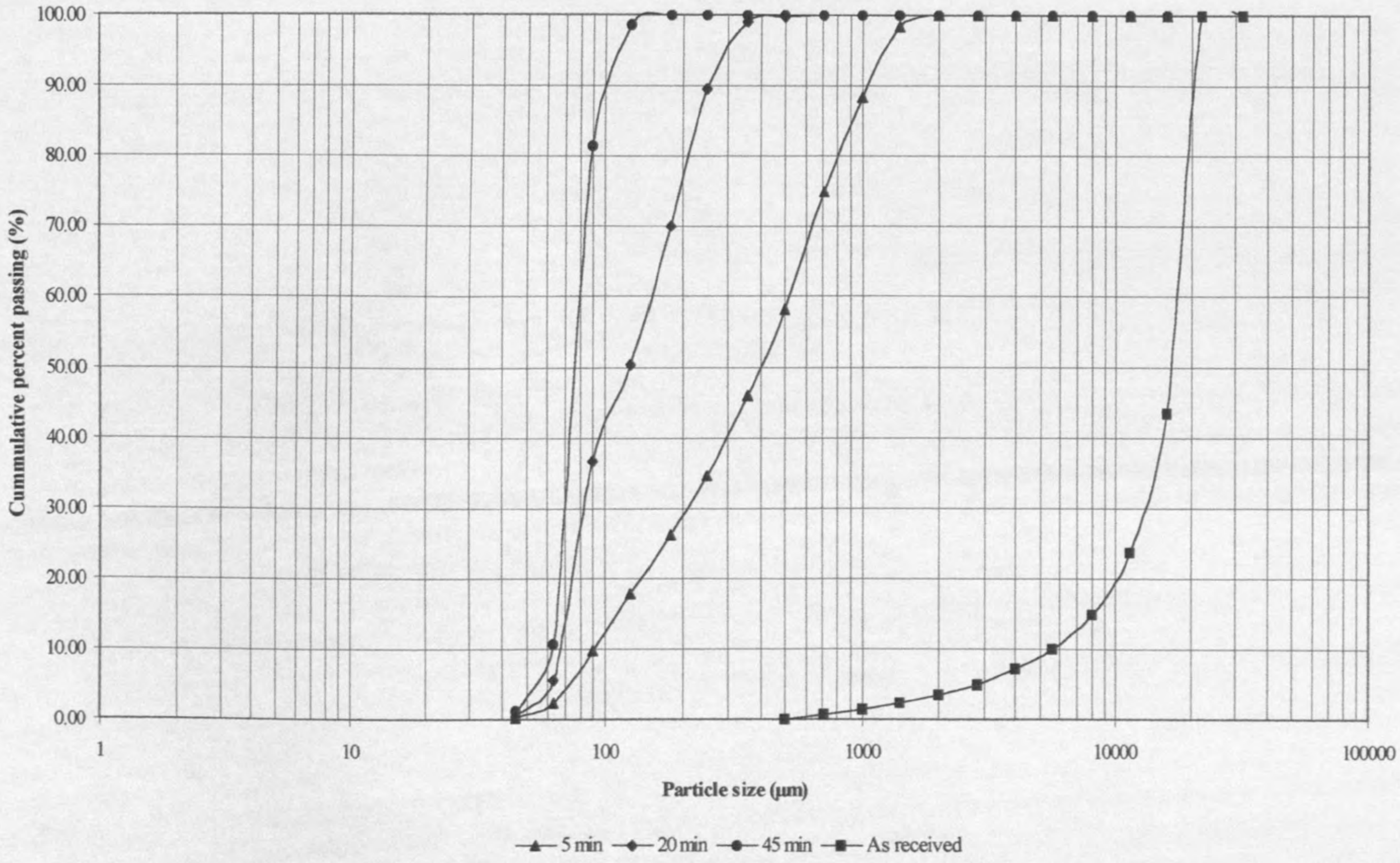


P-7 coal sample B, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Steam addition for 1hr, Microwave input power-0.65kW, applied frequency-2.45GHz,
exposure time-8 minutes

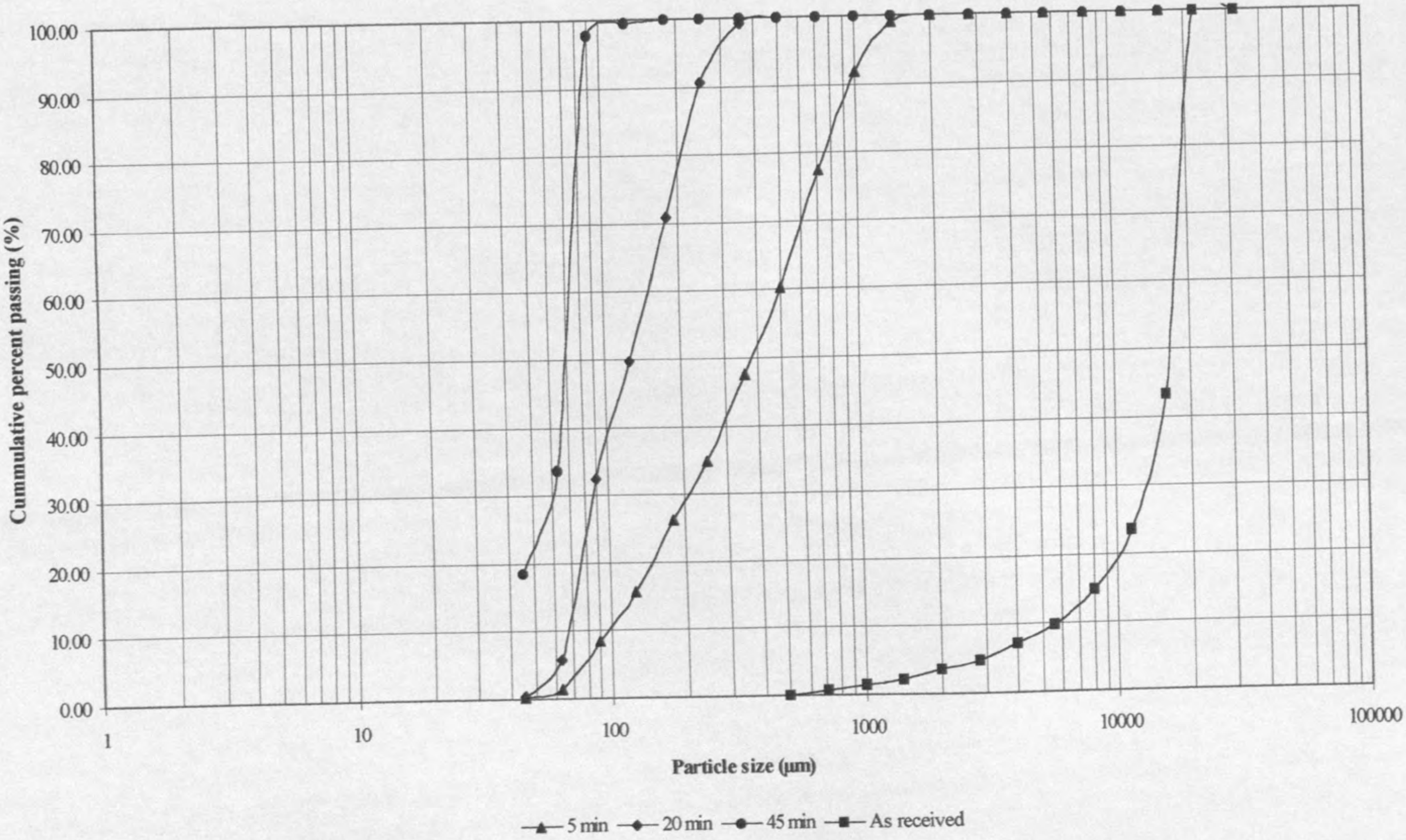


P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

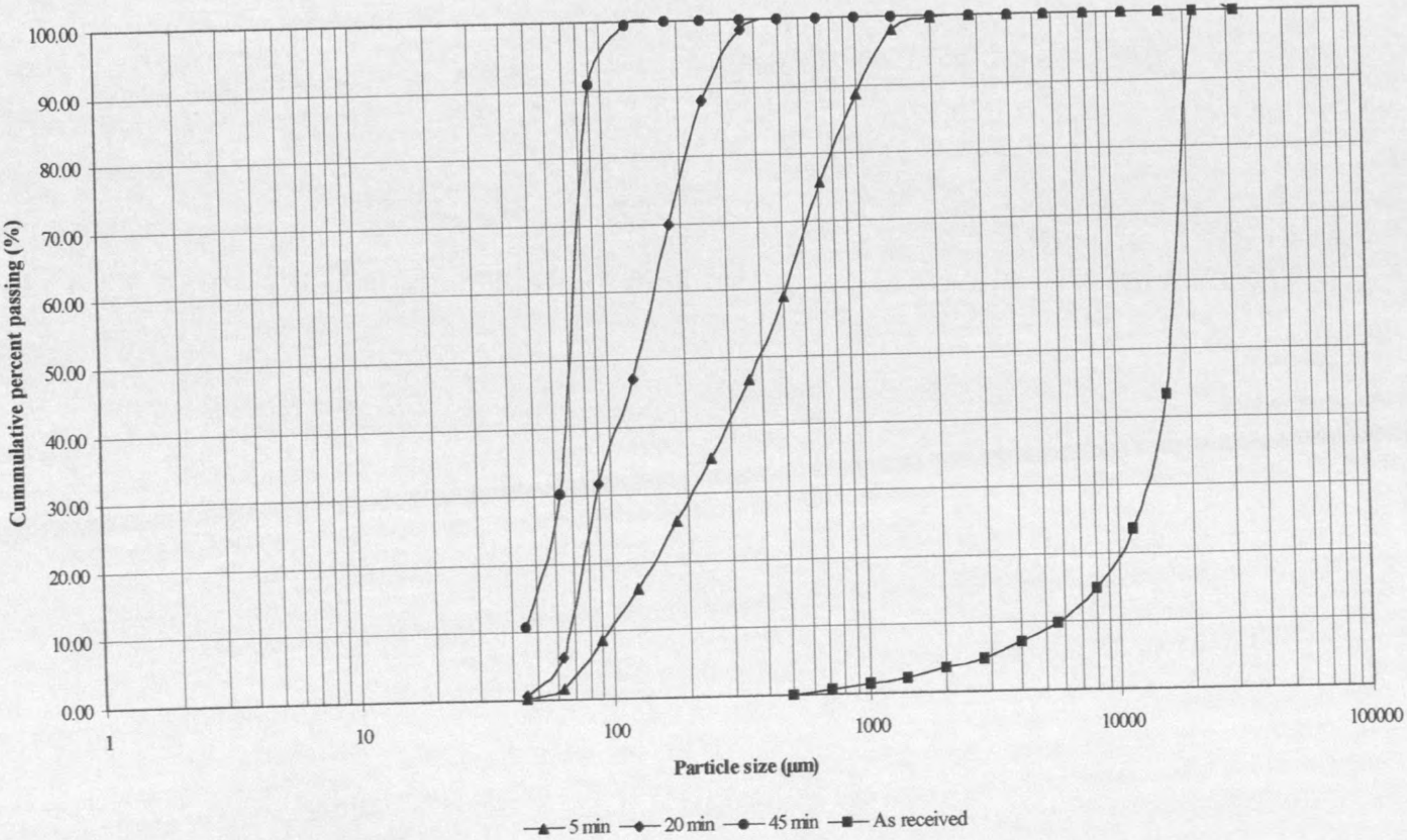
Muffle furnace temperature-110^{0}C 3hr



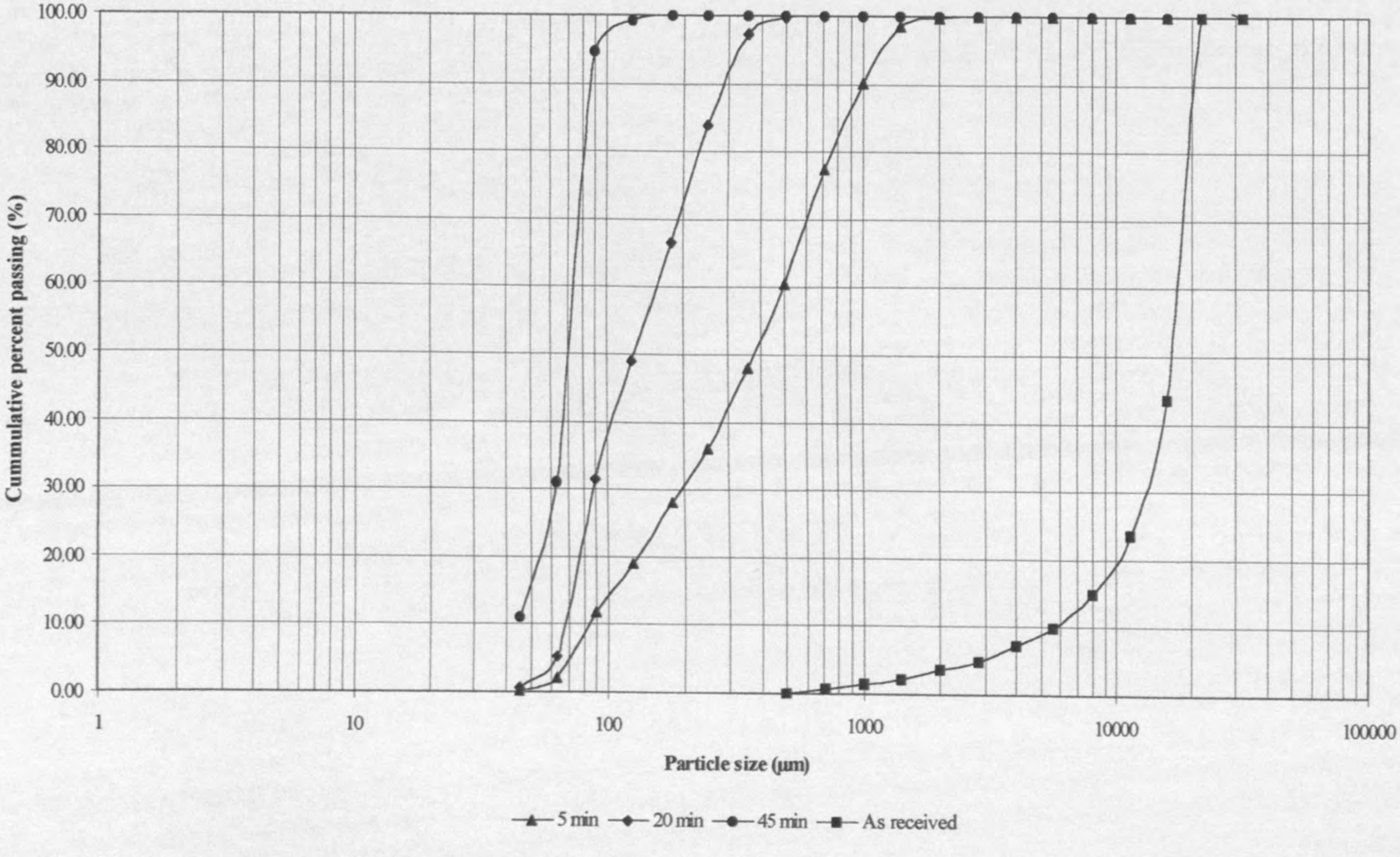
P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



P-7 coal sample A, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water, Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes

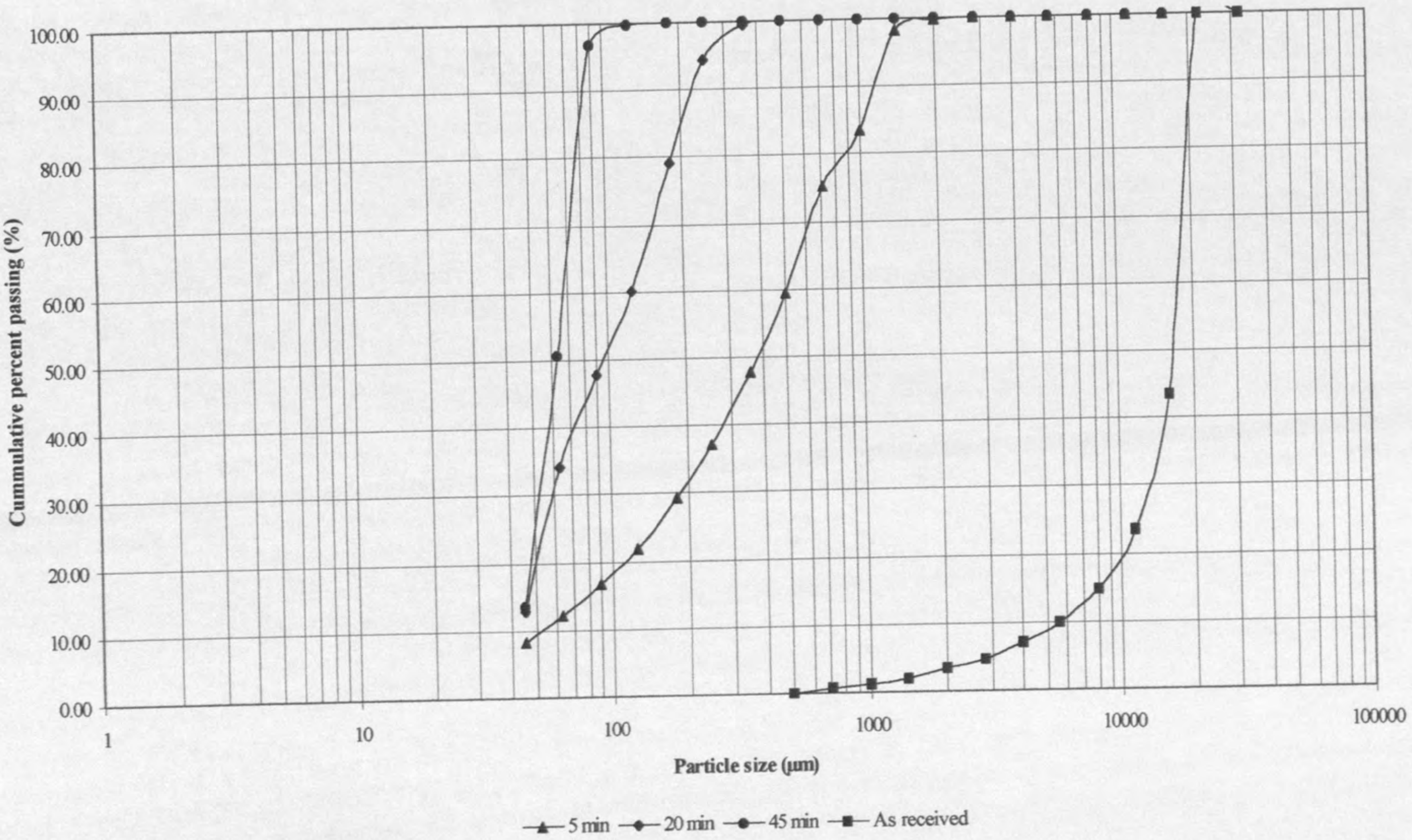


P-7 coal sample B, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water, Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes

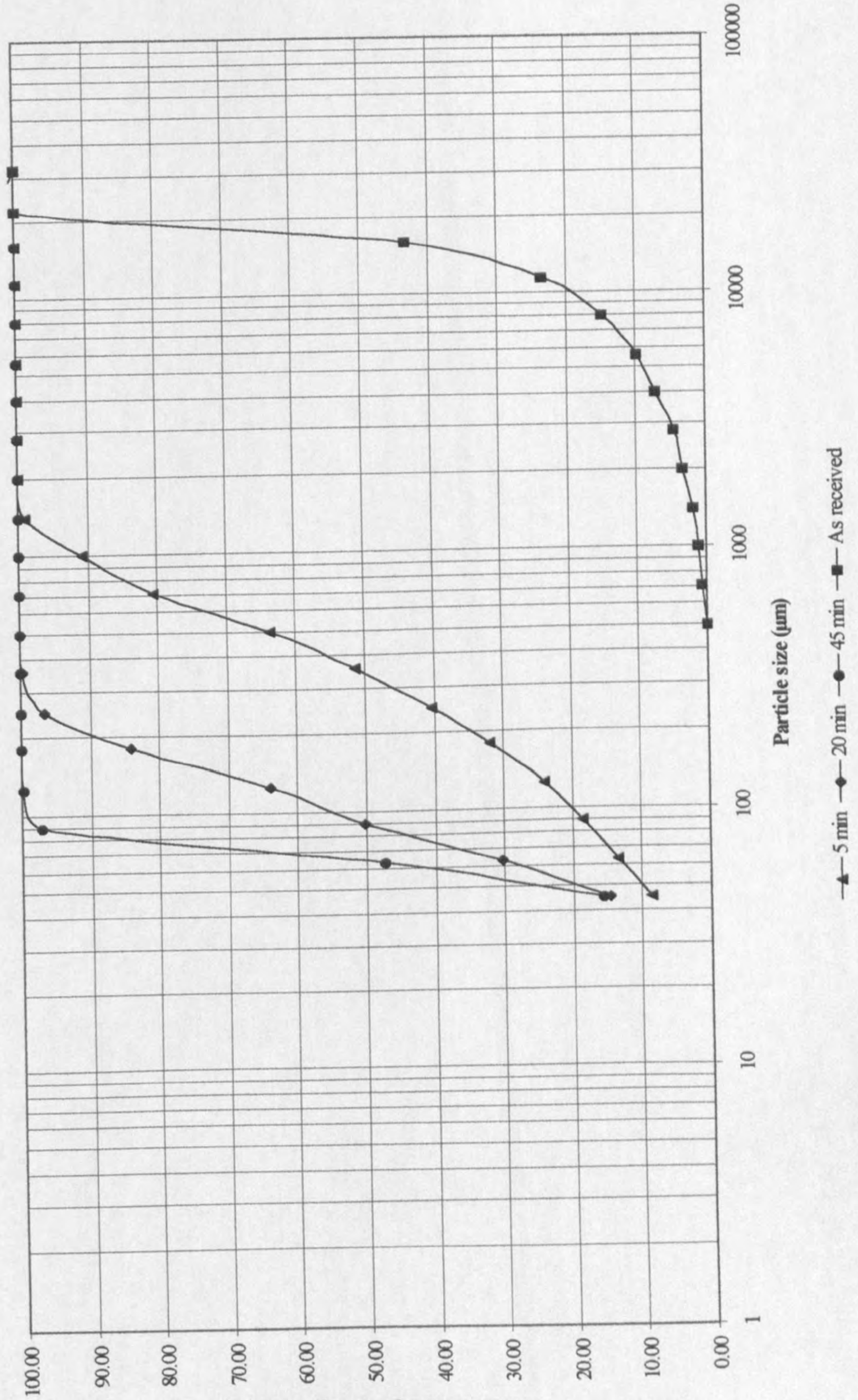


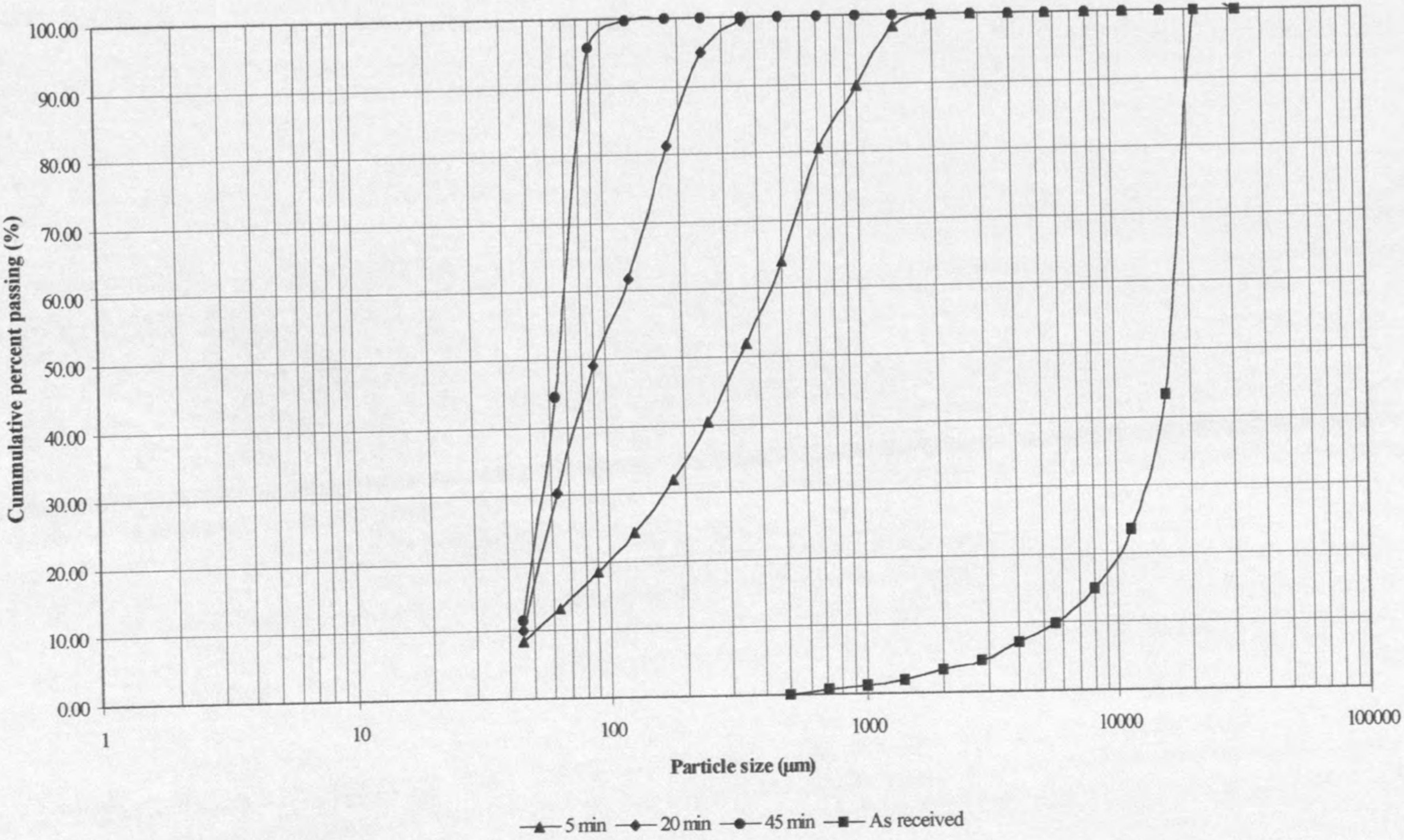
P-7 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

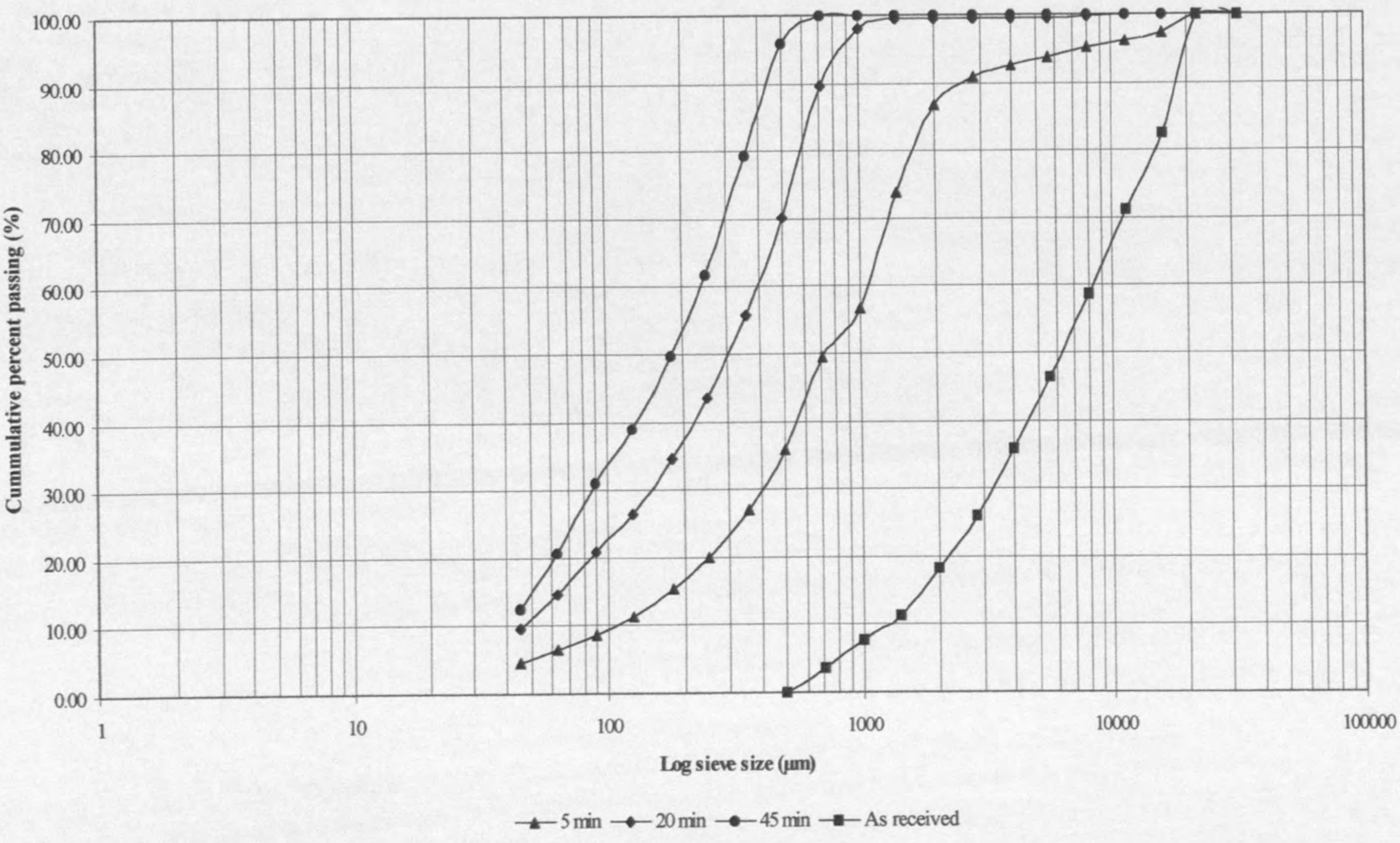
Muffle furnace temperature-110°C 3hr, Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



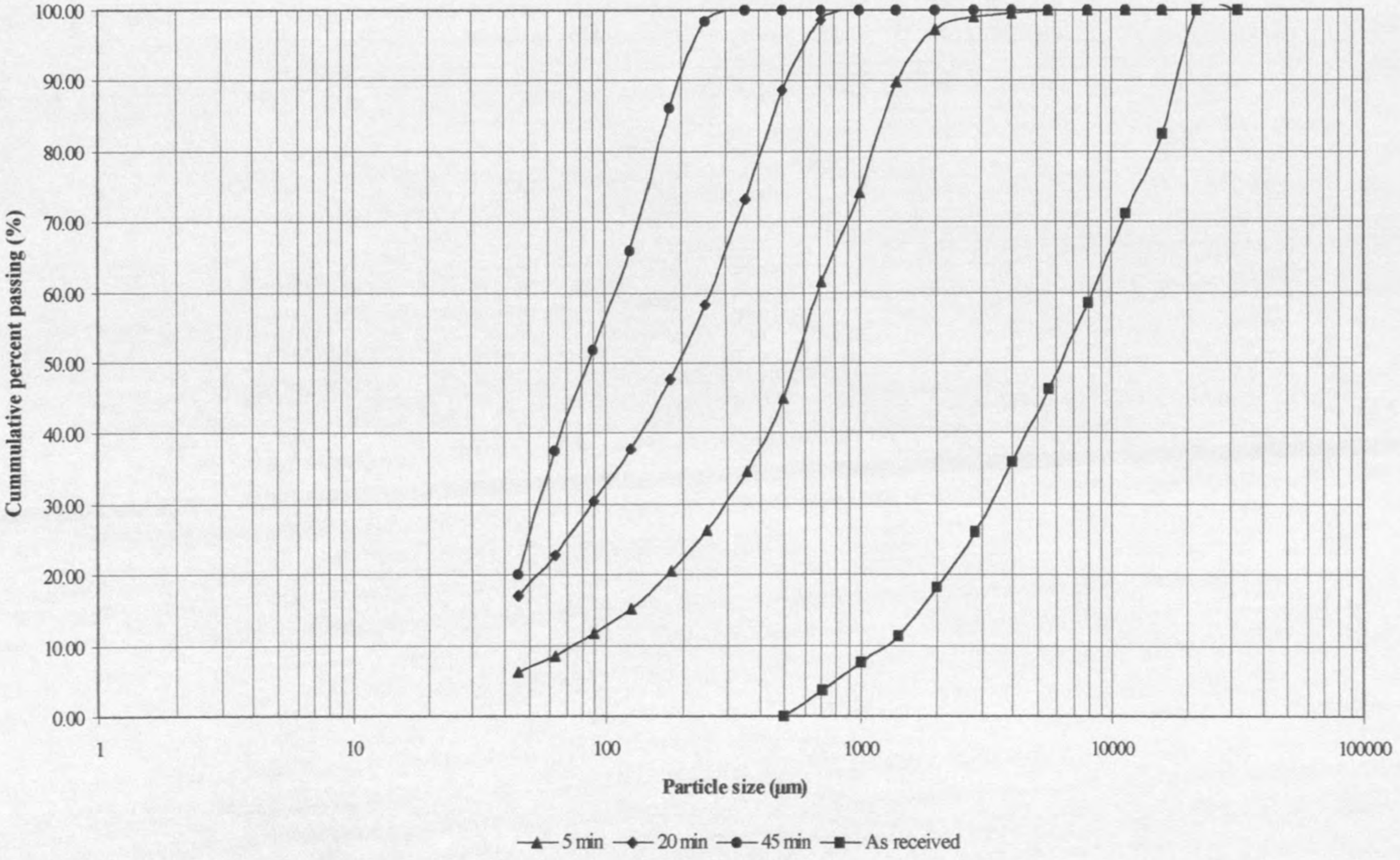
P-7 coal sample A, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water 50g calgon solution, Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes



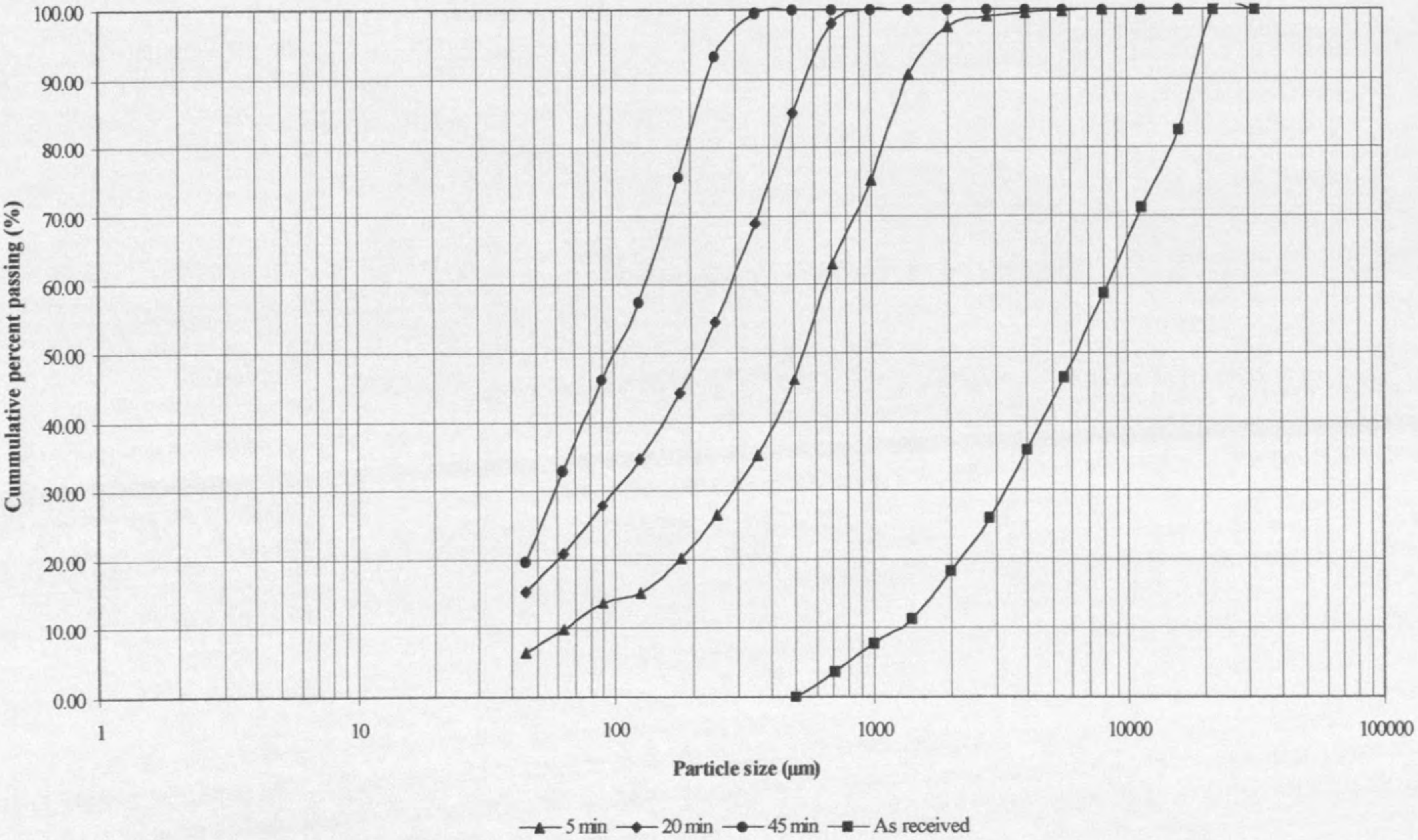

P-7 coal sample B, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water 50g calgon solution, Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-8 minutes


P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

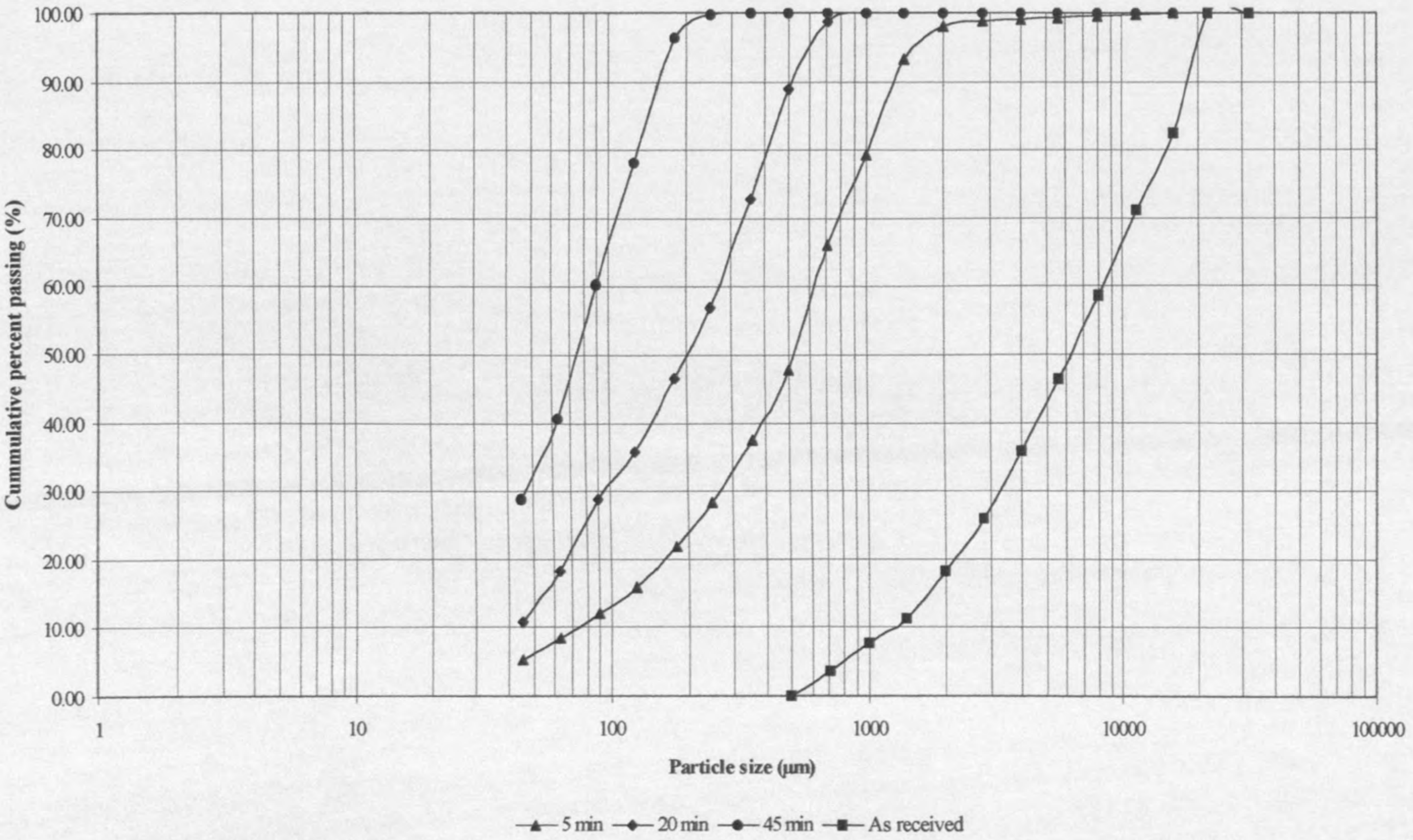
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Muffle furnace temperature-110^0C 3hr



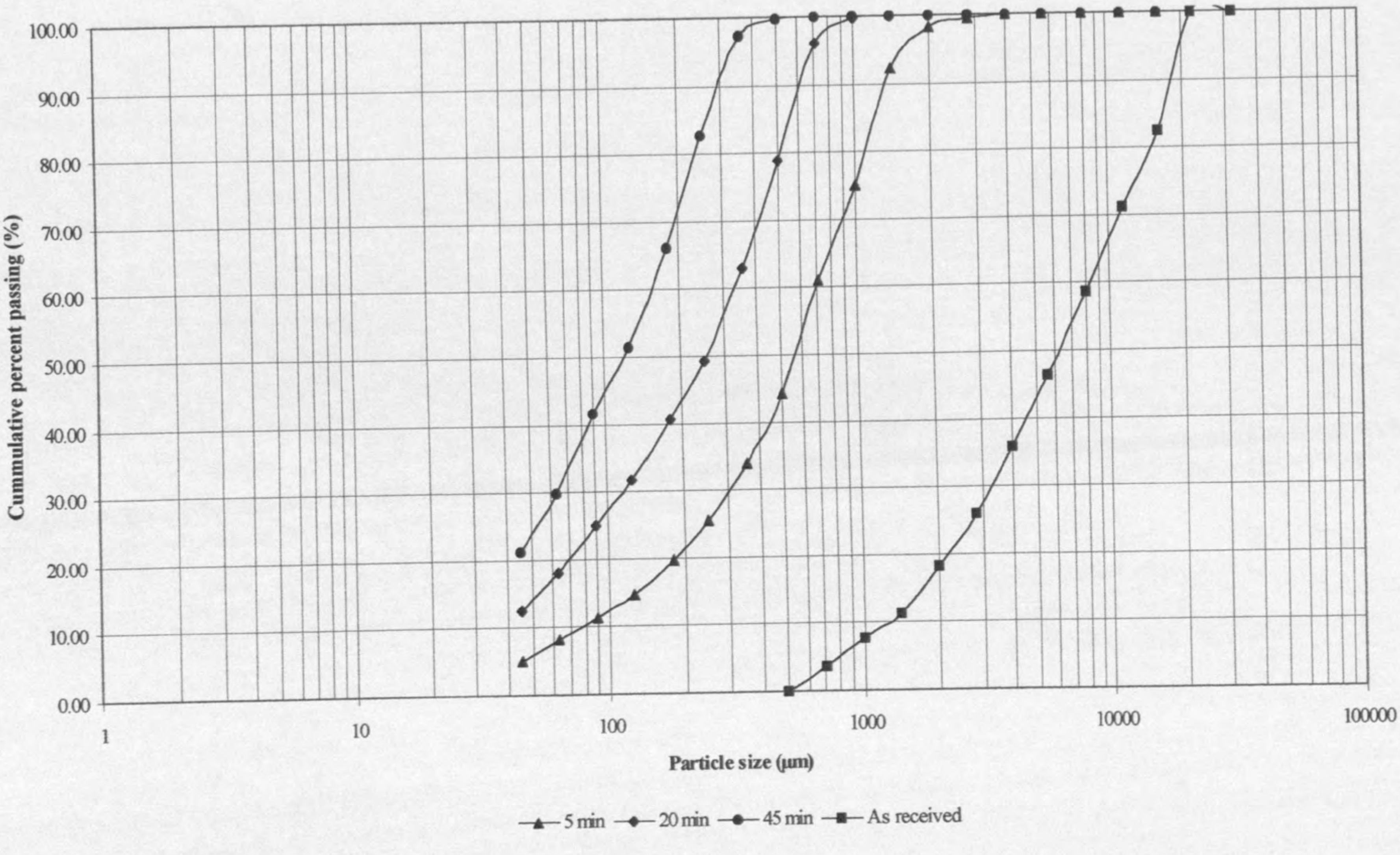
P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwaved input power-0.65kW, applied frequency-2.45GHz, expoure time-8 minutes



P-2 coal sample A, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water, microwaved input power-0.65kW, applied frequency-2.45GHz, expoure time-8 minutes

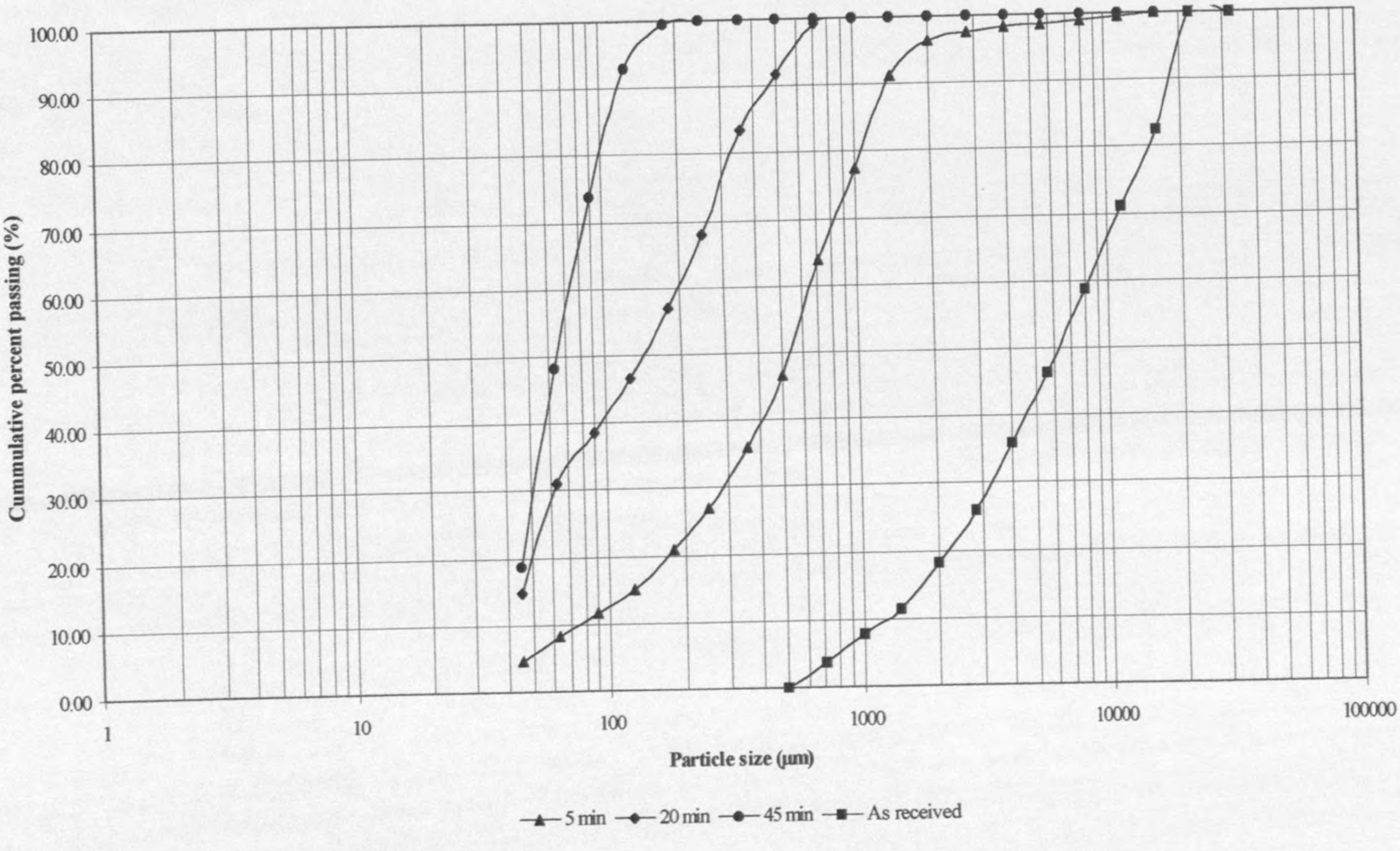


P-2 coal sample B, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Addition of 400ml water, microwaved input power-0.65kW, applied frequency-2.45GHz, expoure time-8 minutes



P-2 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g

Muffle furnace temperature-110^{0}C 3hr,microwaved input power-0.65kW, applied frequency-2.45GHz, expoure time-8 minutes

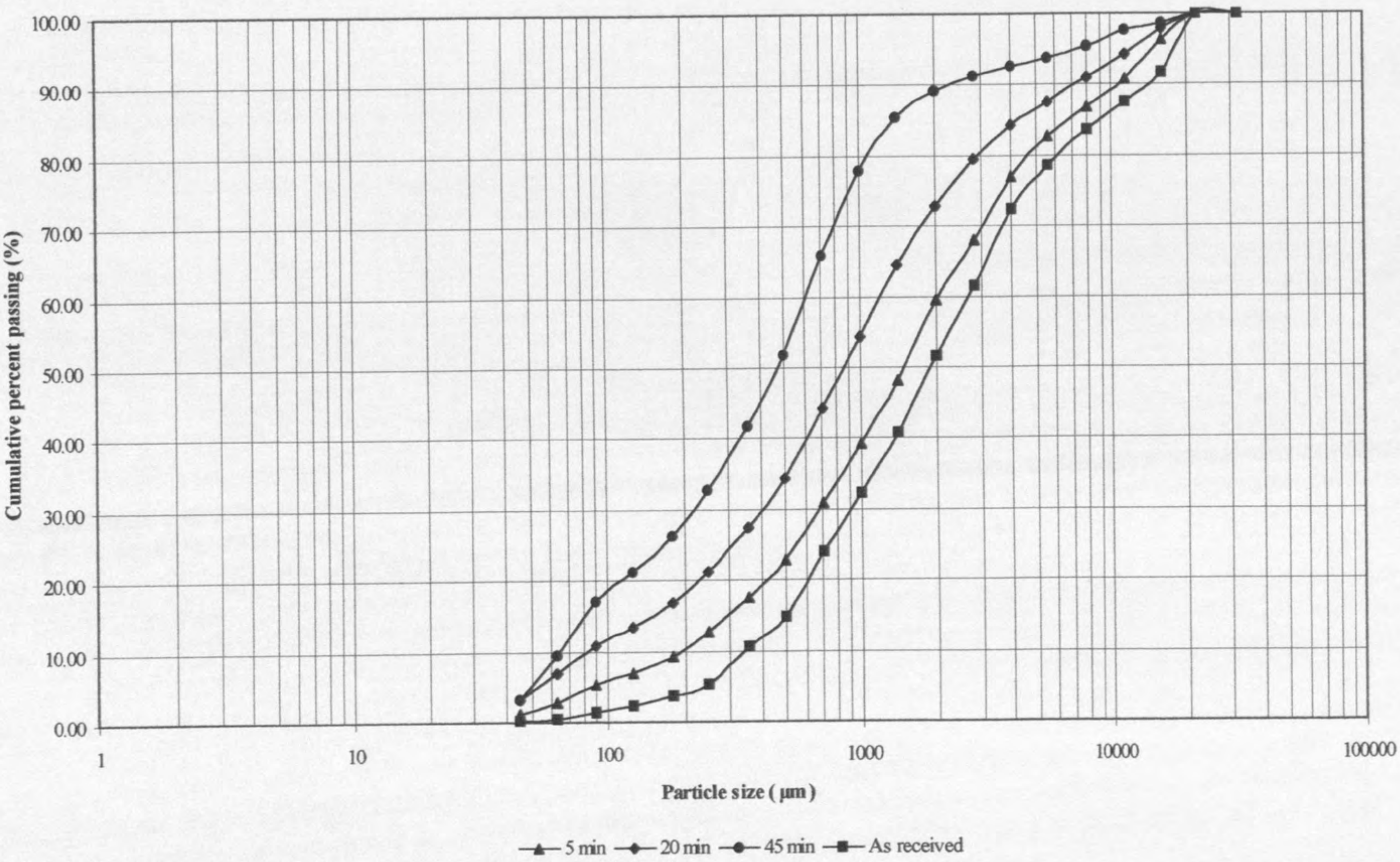


APPENDIX D10

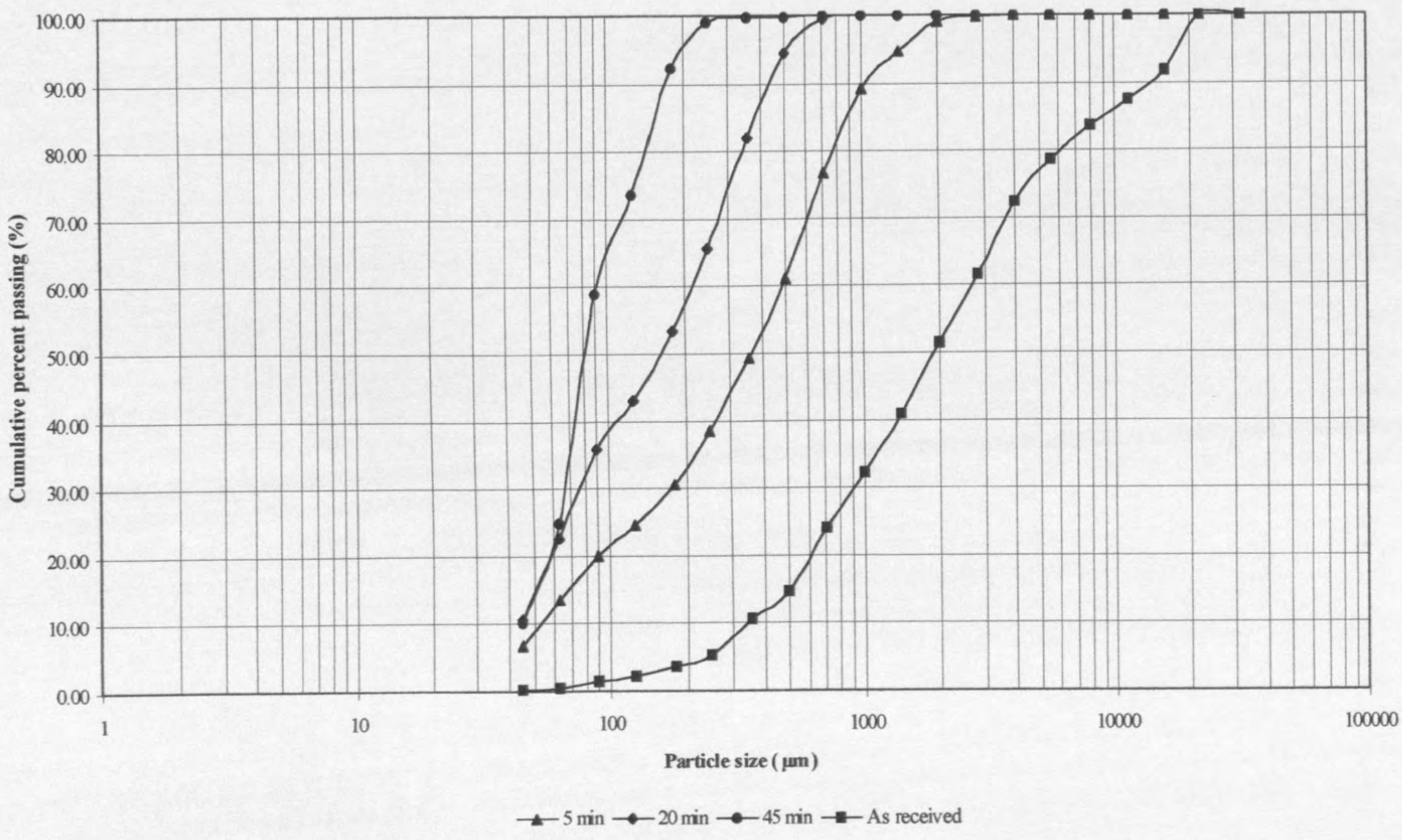
MICROWAVE TREATED COALS PILOT-SCALE PRELIMINARY TESTS ROD MILL SIZE DISTRIBUTIONS

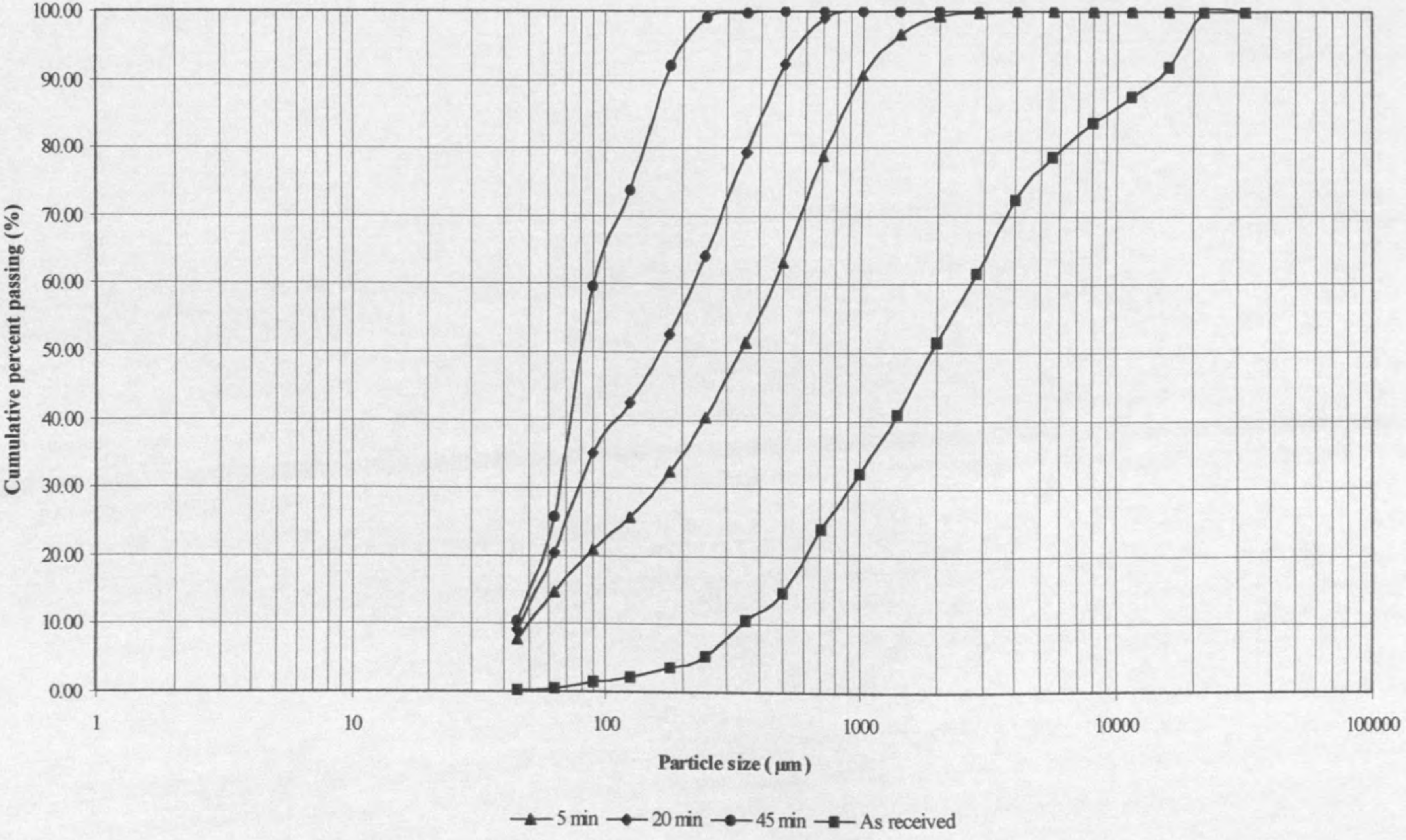
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, load-3kg, Microwave input power-3.3kW, frequency-2.45GHz, exposure time-12 minutes, 220kWh/t, milled as 500g sub-sample D10-13

P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

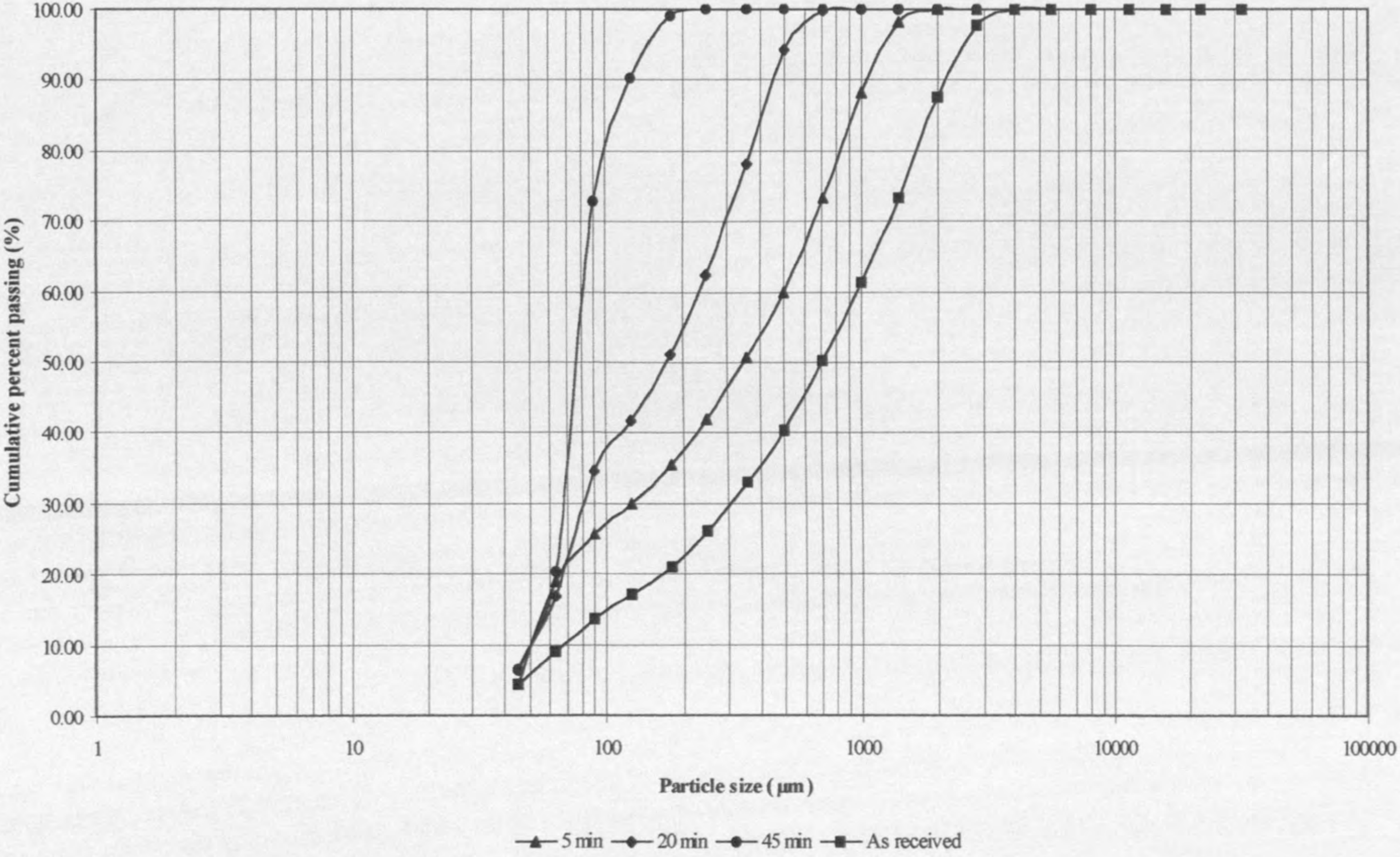


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-10 minute, 220kWh/t



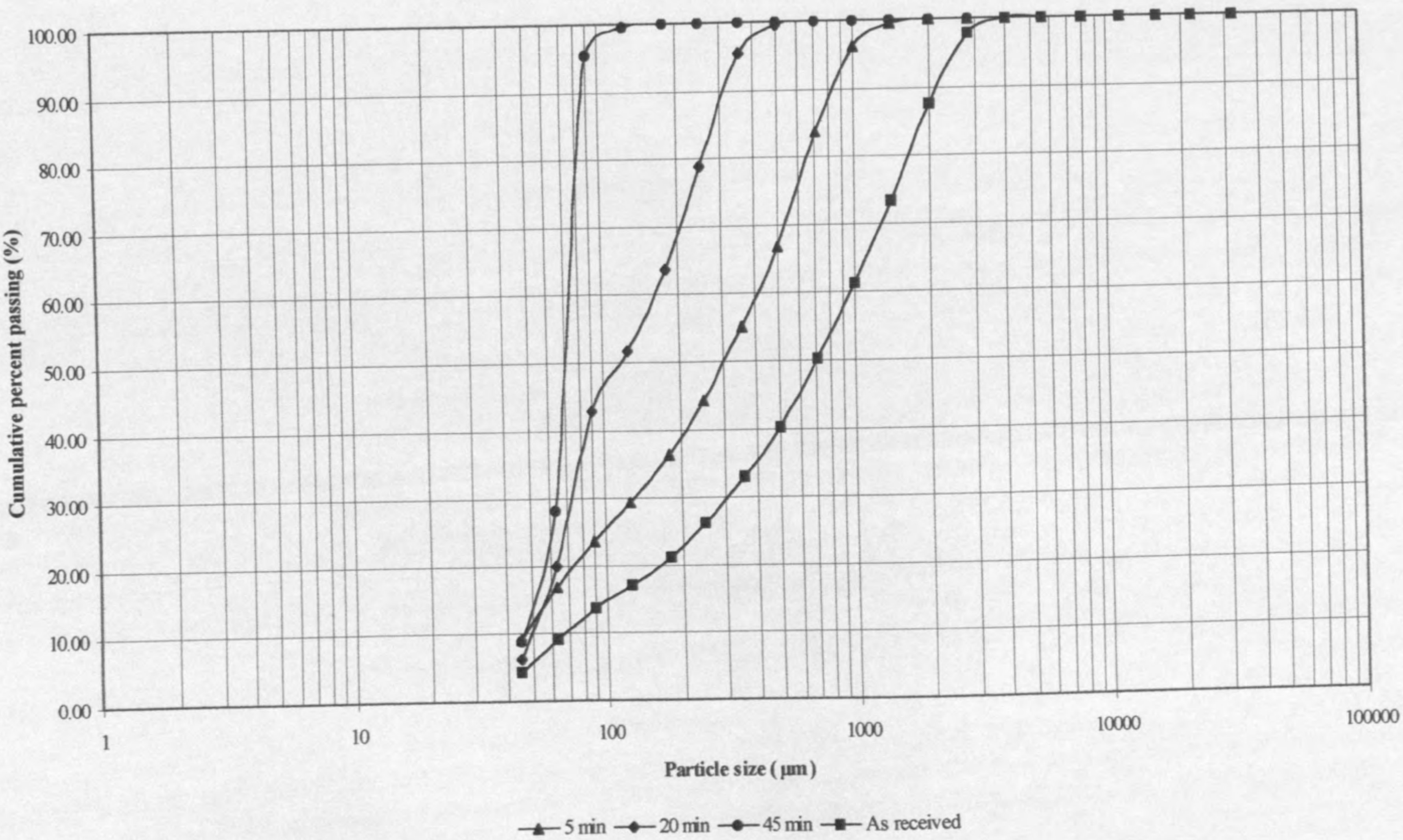


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-2.6kW, applied frequency-2.45GHz, exposure time-152s, 220kWh/t

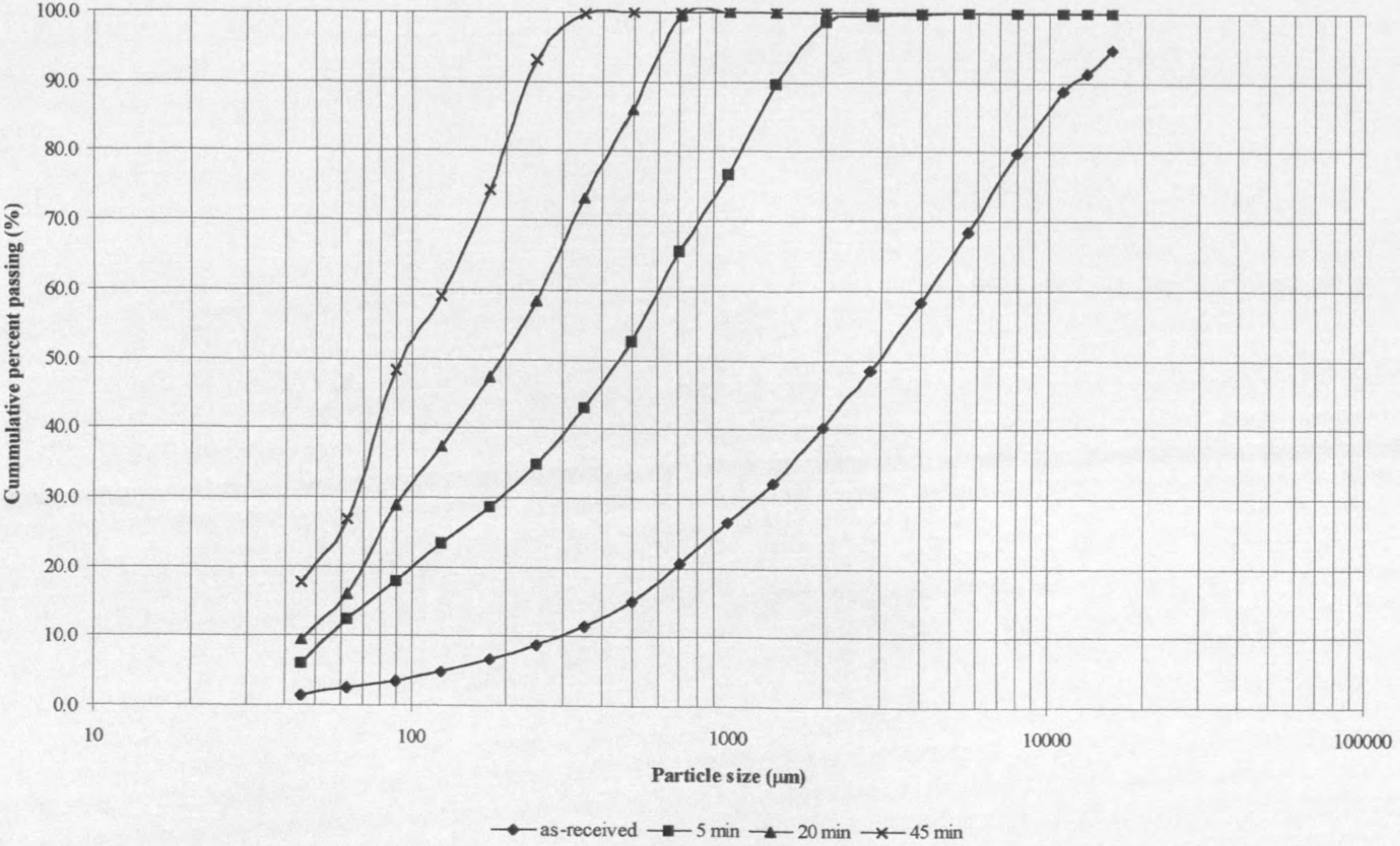


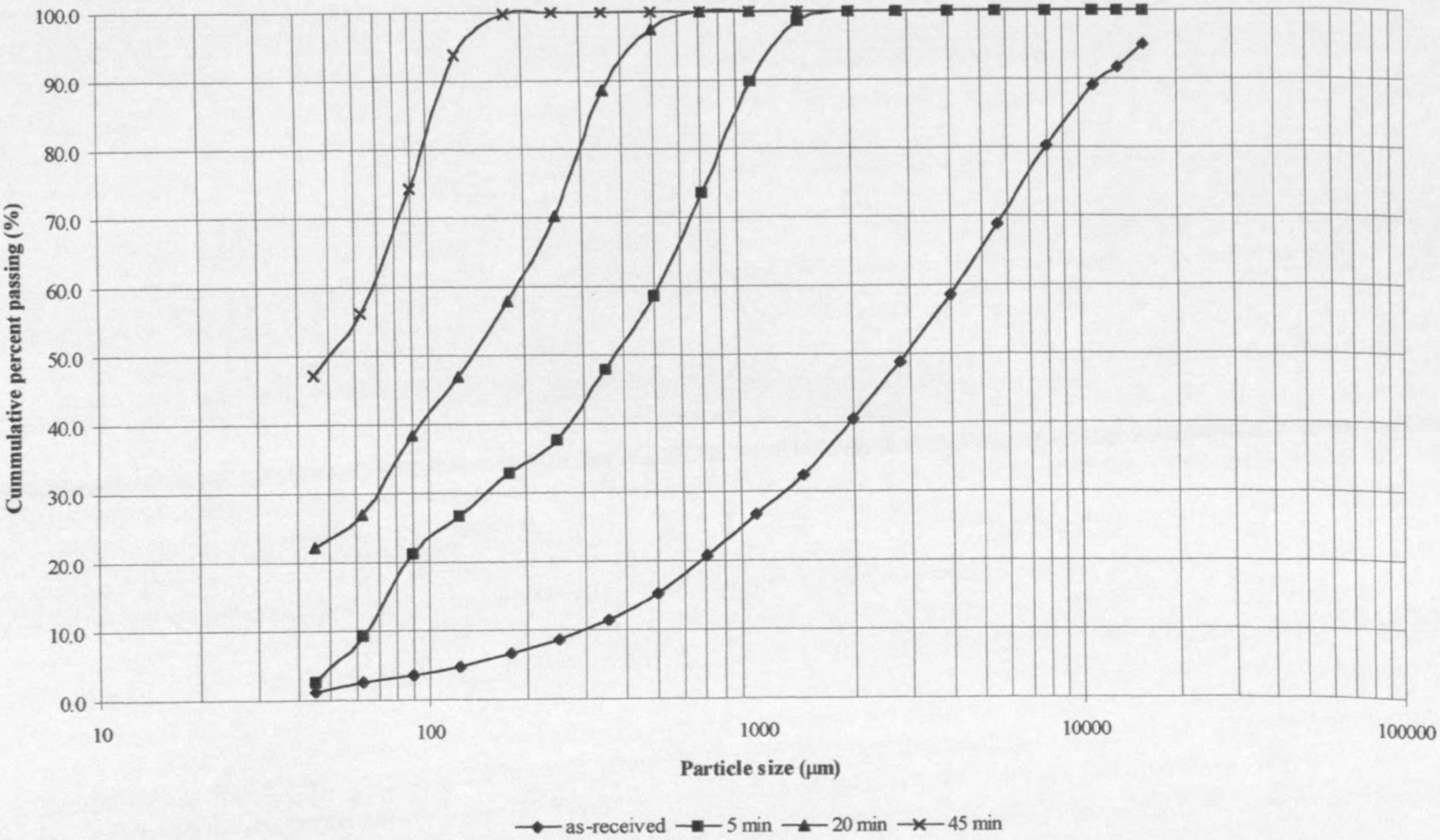
P-6 coal (-3mm), rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received

P-6 coal (-3mm), rod mill, number of rods-7, operating speed-100rpm, sample load-500g
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-10 minute, 220kWh/t



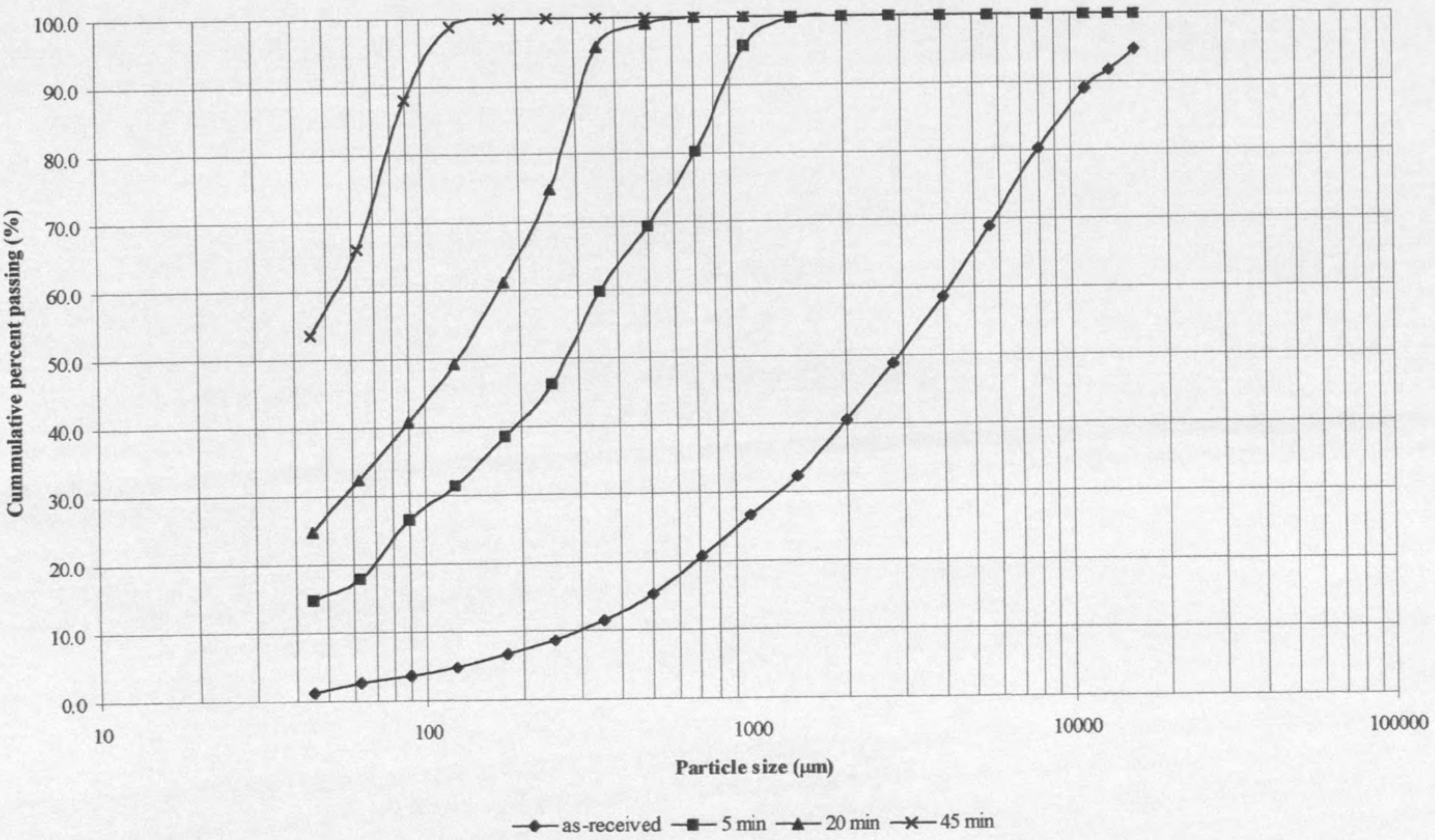
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-500g
As-received



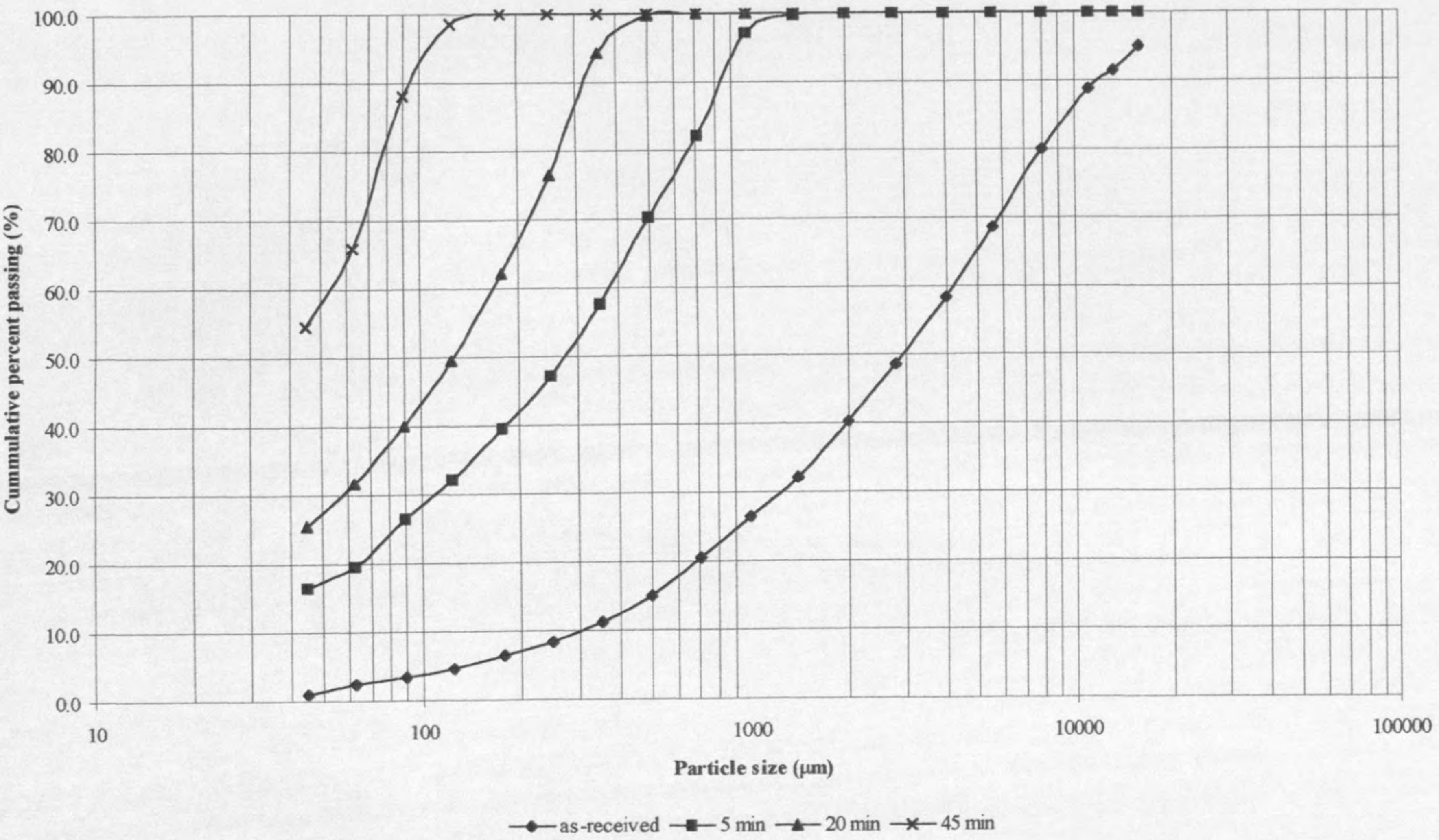


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-1kg
Microwave input power-1.1kW, applied frequancy-2.45GHz, exposure time-8 minutes (220kWh/t)
milled as 500g sub-sample

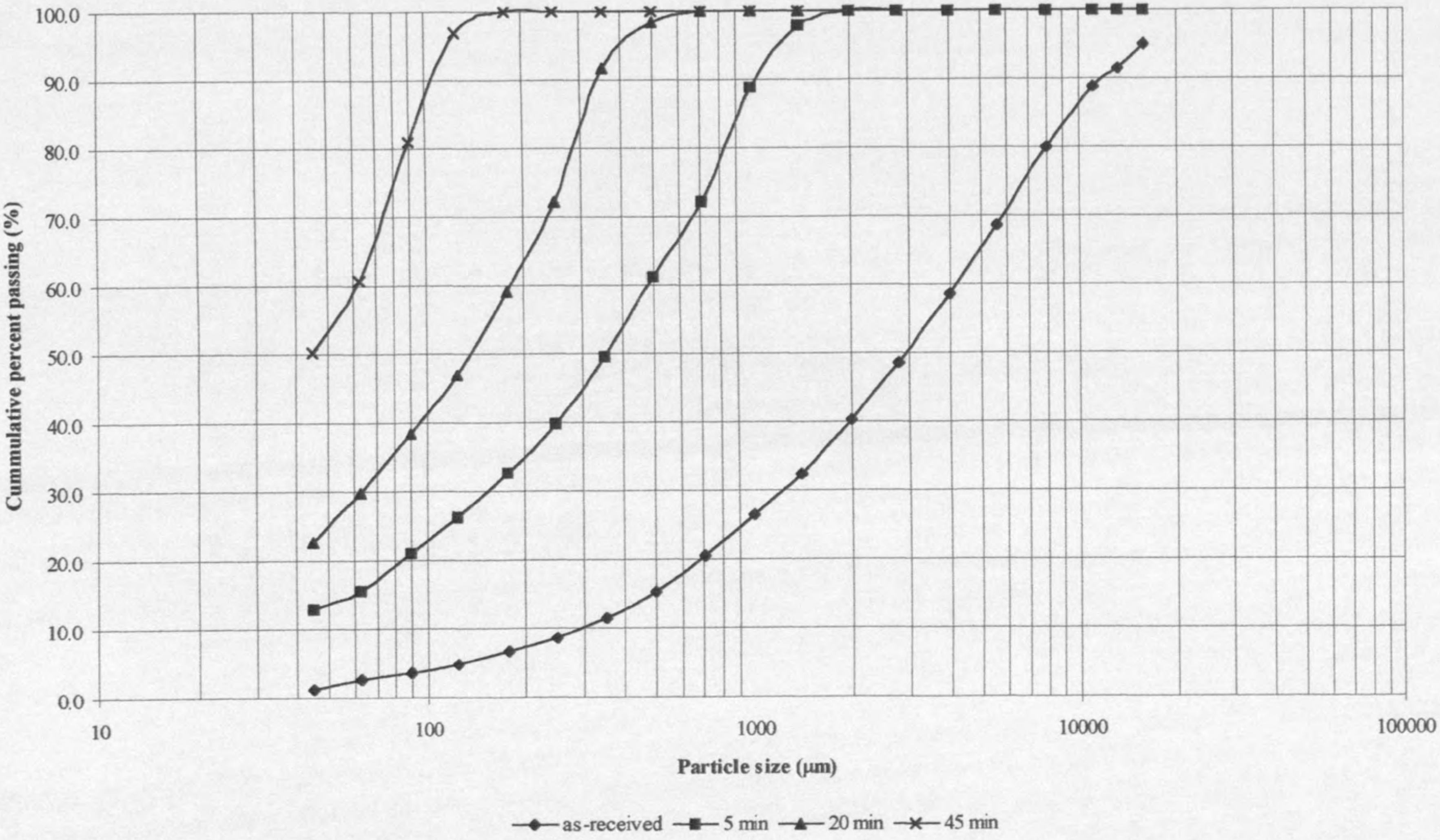
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-1kg
Microwave input power-3.3kW, applied frequancy-2.45GHz, exposure time-4 minutes (220kWh/t)
milled as 500g sub-sample



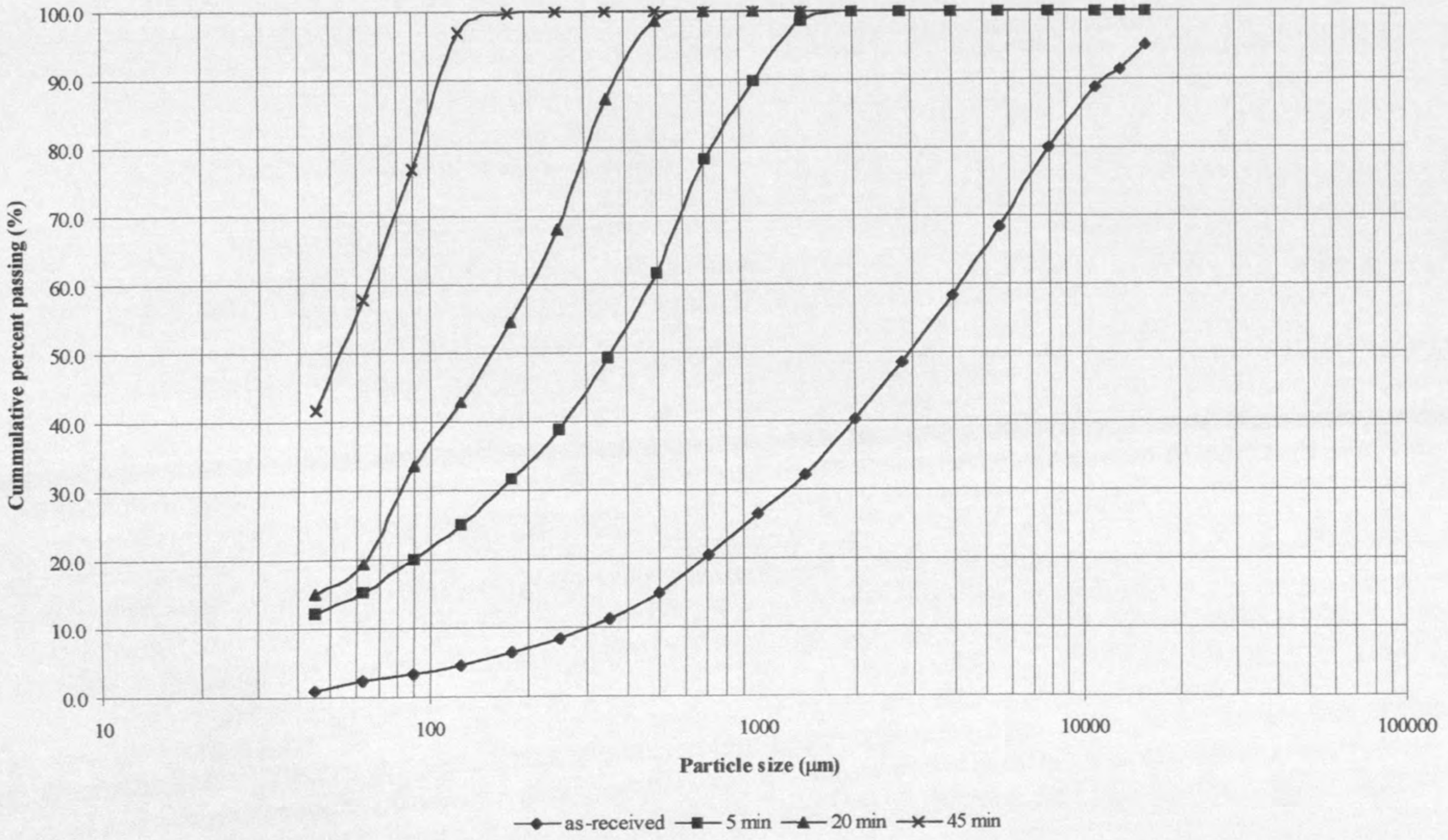
P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-1kg
Microwave input power-6.6kW, applied frequancy-2.45GHz, exposure time-2 minutes (220kWh/t)
milled as 500g sub-sample



P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-2kg
Microwave input power-3.3kW, applied frequancy-2.45GHz, exposure time-8 minutes (220kWh/t)
milled as 500g sub-sample

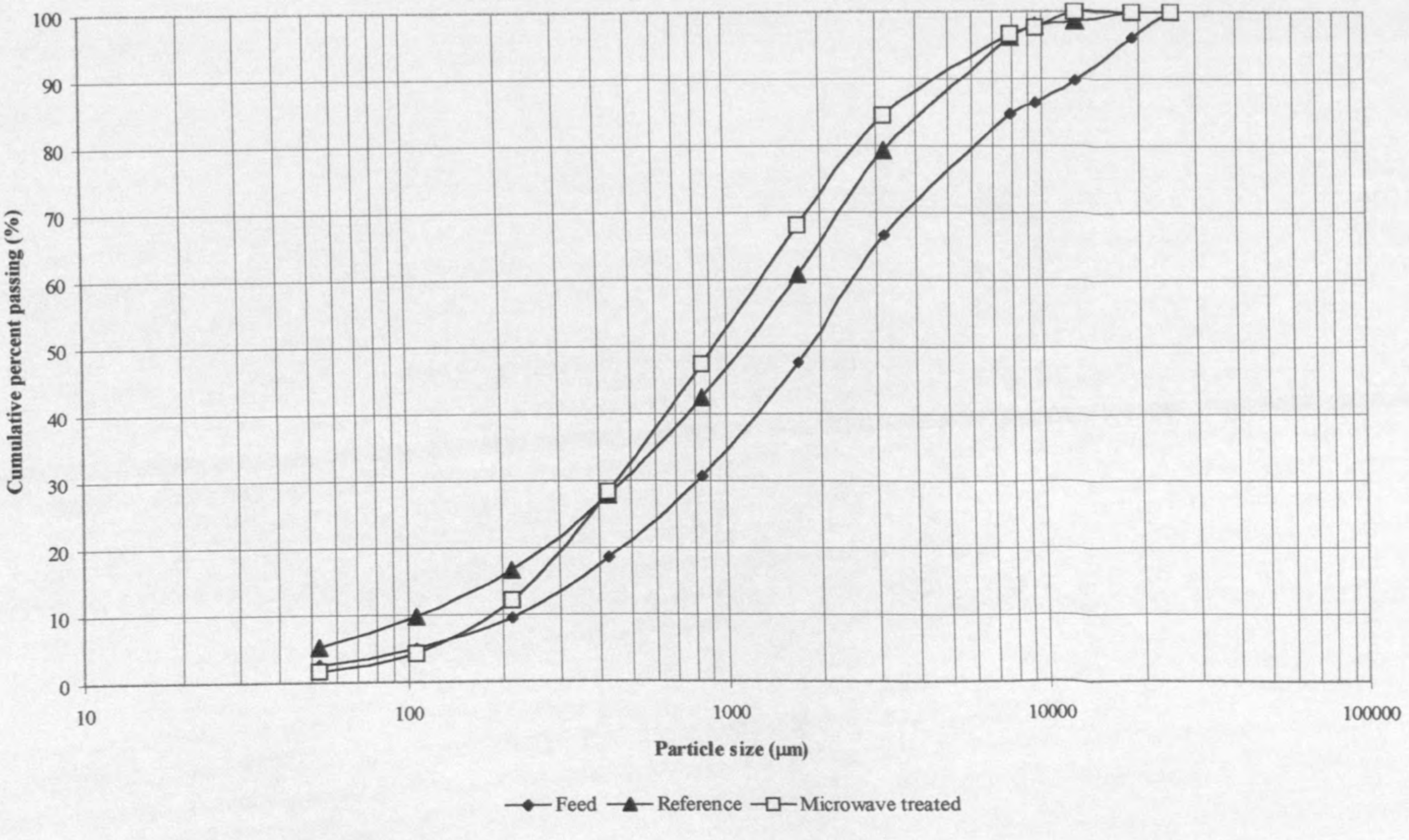


P-5 coal, rod mill, number of rods-7, operating speed-100rpm, sample load-3kg
Microwave input power-3.3kW, applied frequancy-2.45GHz, exposure time-12 minutes (220kWh/t)
milled as 500g sub-sample



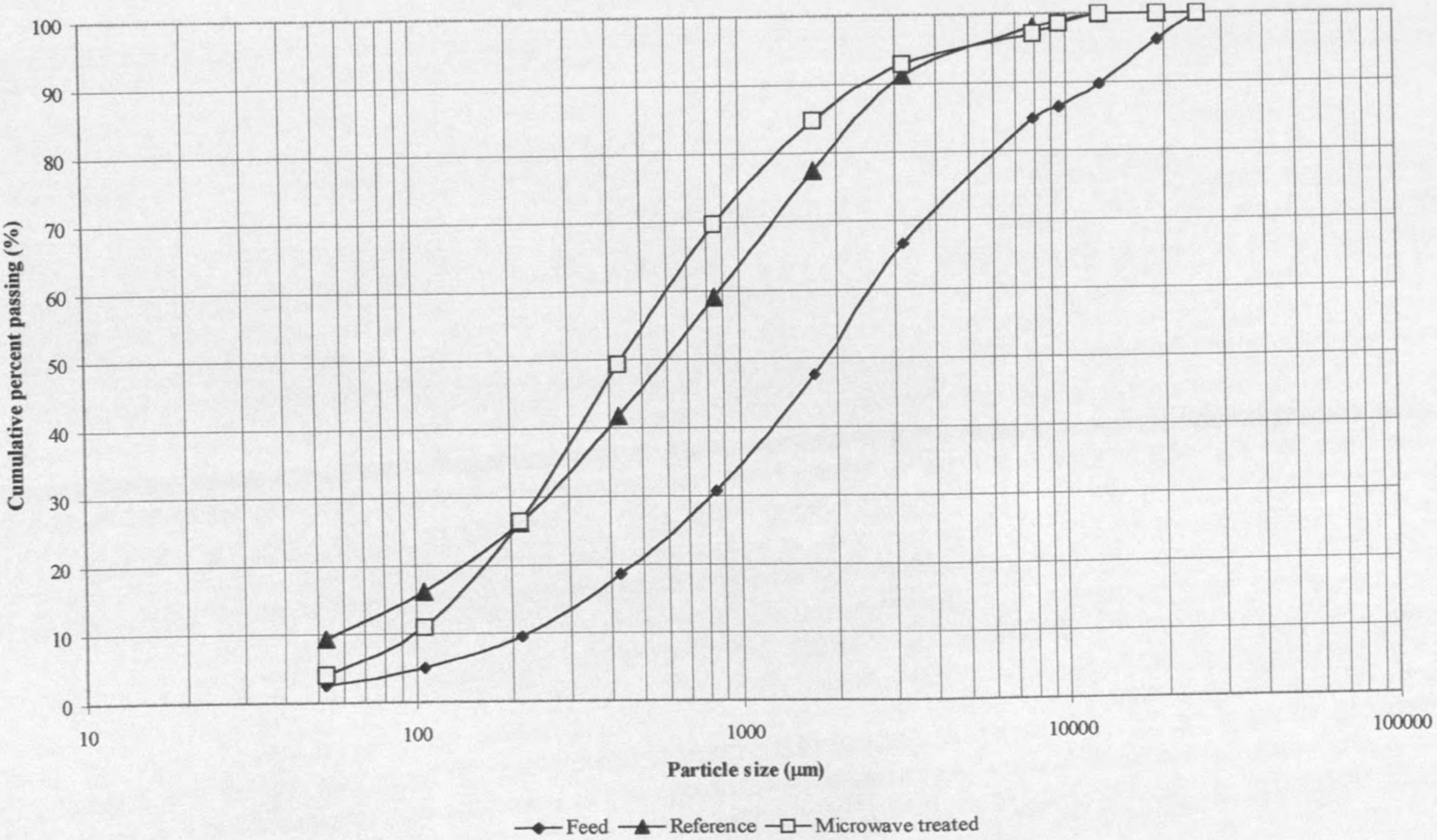
APPENDIX D11

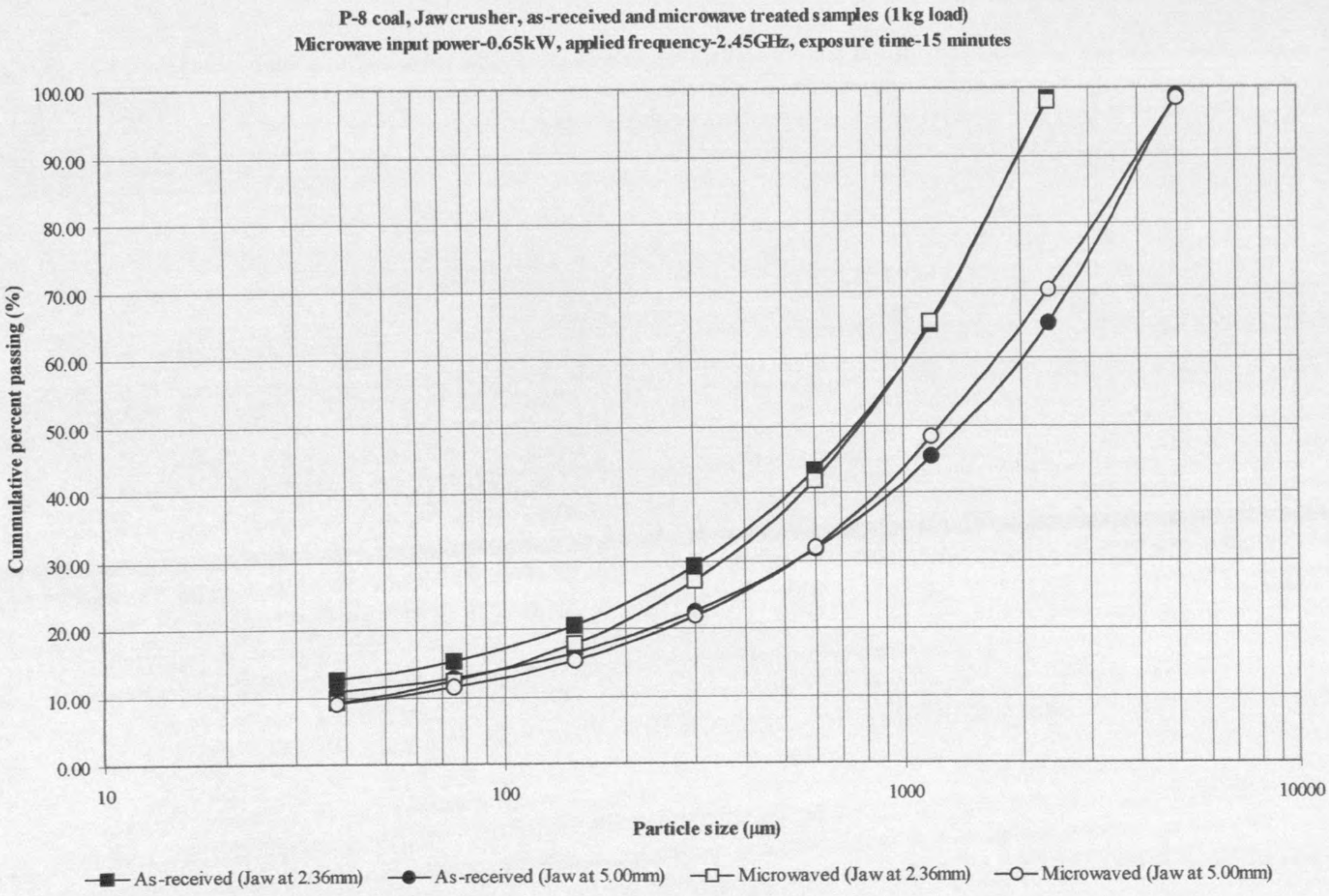
MICROWAVE TREATED COAL PILOT-SCALE TESTS BARMAC MILL, JAW AND ROLL CRUSHER SIZE DISTRIBUTIONS

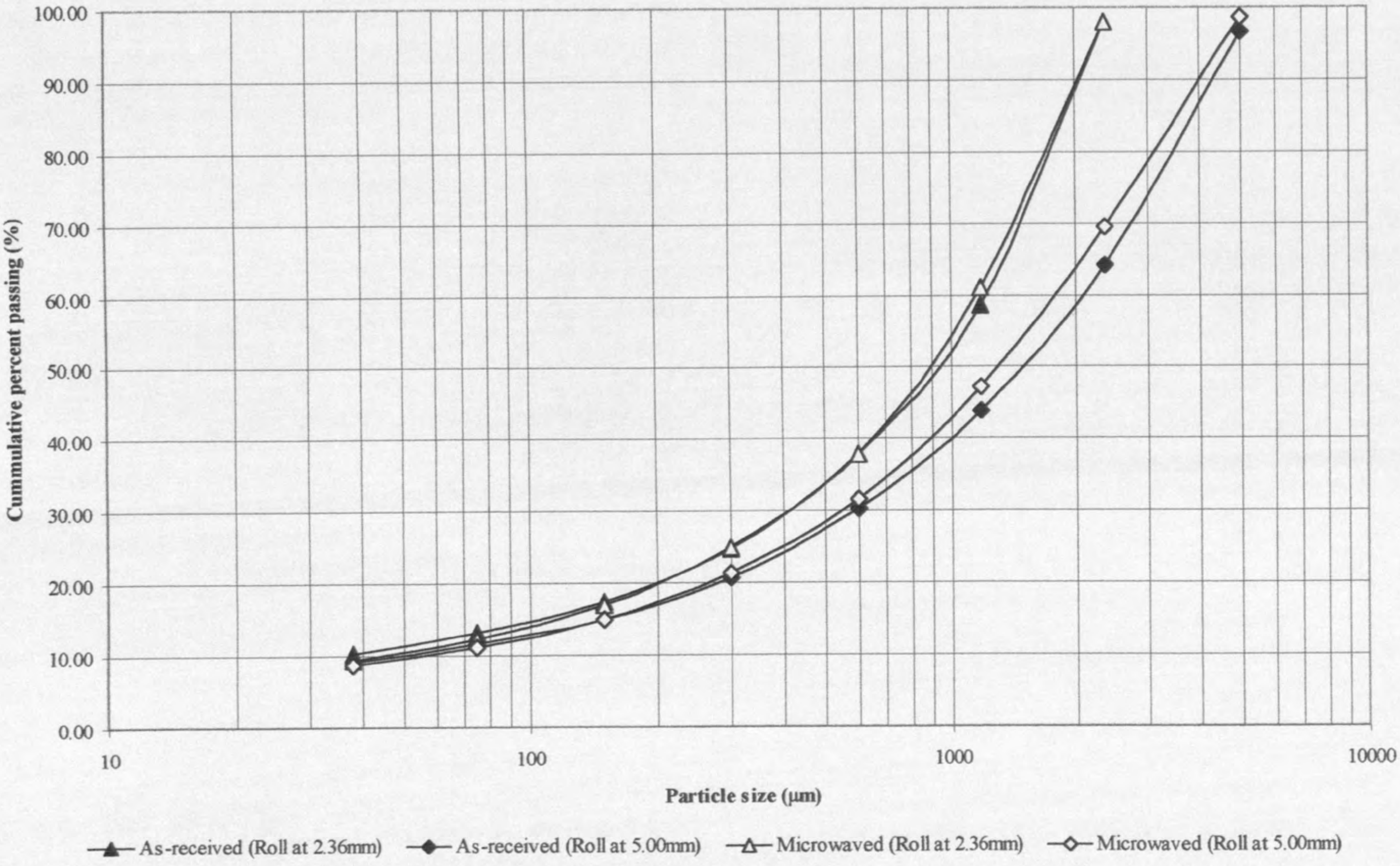


P-8 coal, Barmac Rock on Rock crusher, as-received and microwave treated samples,
operating speed-3000rpm, sample load-250kg
Microwave input power-6.6kW, applied frequency-2.45GHz, 220kWh/t

P-8 coal, Barmac Rock on Rock crusher, as-received and microwave treated samples,
operating speed-5000rpm, sample load-250kg
Microwave input power-6.6kW, applied frequency-2.45GHz, 220kWh/t




P-8 coal, Jaw crusher, as-received and microwave treated samples (1kg load)
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-15 minutes
Cummulative percent passing (%)
100.00
90.00
80.00
70.00
60.00
50.00
40.00
30.00
20.00
10.00
0.00
10
100
1000
10000
Particle size (µm)
As-received (Jaw at 2.36mm)
As-received (Jaw at 5.00mm)
Microwaved (Jaw at 2.36mm)
Microwaved (Jaw at 5.00mm)



P-8 coal, Roll crusher, as-received and microwave treated samples (1kg load)
Microwave input power-0.65kW, applied frequency-2.45GHz, exposure time-15 minutes

APPENDIX E1

DIELECTRIC PROPERTIES OF GROUP 1 AND 2 COALS

SUBMITTED ON CD ROM

APPENDIX E2

HIGH TEMPERATURE DIELECTRIC PROPERTIES OF F-1 COAL

SUBMITTED ON CD ROM

APPENDIX E3

DIELECTRIC PROPERTIES OF MICROWAVE TREATED P-3 COAL

SUBMITTED ON CD ROM

APPENDIX E4

DIELECTRIC PROPERTIES OF SELECTED MINERALS

SUBMITTED ON CD ROM