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**DEVELOPMENT OF WATER-BASED CORE TECHNOLOGY
FOR LIGHT ALLOYS**

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requirements of the University of Teesside
for the degree of Doctor of Philosophy

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collaboration with Casting Solutions, Sheffield.

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ABSTRACT

This thesis describes, in a manufacturing context, the development of new water-based core technology for light alloys.

Cores used for steel casting are made from fused silica and are removed using hot sodium hydroxide under refluxing (pressurising hot acids). However, aluminium and other light alloys are attacked by sodium hydroxide. Currently there is no good core system for aluminium and other light alloys. It is therefore desirable to find an alternative material/leaching agent combination for casting aluminium and other light alloys.

The recent research review has shown that ceramic cores are mostly made by fused silica with different additives. The previous research has suggested using fused silica (different mesh size) as filling material and using magnesium oxide to control the slurry working life of core mixes. Calcium silicate assists core leaching in dilute acid. The plaster (calcium sulphate) in the form of proprietary plasters (Crystalcal R, Fine Casting Plaster) is used to create bond and gives strength to the core. Lithium carbonate acts as an accelerator, improving the strengthening effect of the plaster in the cores. The binder (Ludox[®] AM) and water act (as added materials) to bind the composition

Core compositions were made with different core trials to produce a core, which records suitable strength and quick leaching properties for light alloys. Core trials were individually mixed and poured into a wooden core box. Cores were pre-dried for twenty-four hours. Cores were fired to different temperatures for two hours, followed

by two hours cooling. The cores were subjected to computerised three-point bend test to record the Modulus of rupture (MOR). The plain strain fracture toughness and Weibull parameters were calculated. The Weibull parameter was plotted using Minitab analysis software. Using the cores, gravity die casting process were carried out. The subsequent castings was dipped in diluted nitric, citric and acetic acid to leach out the core.

Using different core compositions, core trials were mixed, poured, dried, tested and leached. The high amount of plaster in core trials records high MOR when cores were fired between 200°- 400° C and the opposite result when fired between 600°- 800° C. The different grade of plasters(CRP,FCP) do not influence the strength. One percent of magnesium oxide gives a very short working life. High amount of binder(Ludox[®] AM) in core produce strong cores.

Workable MOR results can be obtained depending on composition allowing manual handling or a waxing process. The fracture toughness is typical of a brittle material, with matching Weibull parameters. The casting process suggests that the new materials are sufficiently refractory. The cores are leached out using diluted nitric, acetic and citric acid at rates compatible with commercial manufacture.

This methodology has successfully produced a core using fused silica with plaster and magnesium oxide for aluminium and possibly for light alloys. Different core trials can be used depending on the specific industrial application relating to strength and removal with acid attacking the metal. Further work is needed to fine tune optimum leaching conditions.

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1.0 INTRODUCTION

1.1 CERAMIC CORES

A core is a pre-formed shape produced in bonded sand or ceramics used to fashion internal features in castings. The core is located in the mould and metal is poured in, filling the mould and surrounding the core. After the metal has cooled and solidified, the core is removed from the casting by various methods.

Ceramic cores are used extensively in the manufacturing of cast engine components, blocks, cylinder heads and a wide range of other engineering and consumer products. Specialist precision applications⁽¹⁾ include the formation of internal cooling passages in cast blades and vanes. The most widely used core material is fused silica because of its very low thermal expansion and it can be leached out of the casting, using aqueous or fused potassium or sodium hydroxide. High temperature deformation of silica has proved problematic, as cores have become more complex. Ceramic cores can produce cavities and holes with a high degree of accuracy and good surface finish.

Cores are prepared by a variety of methods. Sand cores are manufactured using similar methods to sand moulds. Some cores are injection moulded whilst many are prepared simply by slip casting fast-setting ceramic⁽²⁾ slurry or plaster into a core box. The core is then extracted from the mould; whatever the method of manufacture it is then either cured (sand) or fired before use.

In the process of slip casting, quick setting slurries are formed in conventional foundry patterns or in standard core boxes made of wood, plastic, plaster, or metal.

Setup and equipment costs can be quite modest, particularly when integrated into an existing sand operation.

1.2 CASTING WITH CERAMIC CORES

The use of cores in castings can allow the design engineer a degree of flexibility whilst producing cost-effective components. However, the use of cores may bring with it technical problems and additional costs. For an impeller mould, for example, uniform filling around very thin ceramic cores ⁽³⁾ is important to avoid movement or flotation of the core. Although complexity of the casting is enhanced and difficult to machine, features may be incorporated, requiring little or no finish machining; the use of cores is often a compromise in one way or another.

Manufacture of a correctly dimensioned core of appropriate strength is only part of the process. It is necessary that the core is stable during the casting process. The gases generated within the core during casting must be capable of being vented to the outside of the mould, precluding gas porosity and the defect known as a core blow. Complex cores may further be strengthened with metal rods and wires.

It is also essential that the core can be removed without adversely affecting the casting. This can be achieved using a suitable reagent that degrades or dissolves the core. Alternatively, it may be possible to use materials that cause breakdown at an appropriate temperature. A core for a light aluminium casting must degrade at lower temperatures, compared with the temperatures that a core must withstand with e.g steel, which is cast at much higher temperatures.

1.3 AIMS OF THE PROJECT

The basic chemistry of most core materials relies on the Si-O bond in its different manifestations. This may be present in the filler material such as fused silica, a commonly used non-crystalline^(3, 4) (glassy) form of silicon dioxide or in the colloidal silica binder. The latter, a stable dispersion of amorphous silica particles sometimes referred to as a sol or silica sol (e.g. by various Ludox™ brands) is widely used as a binder.

Irrespective of whether the core is made up from a calcium silicate, plaster or a more highly refractory silica composition, the virtue of these materials is that they can be removed from the casting by aqueous refluxing sodium hydroxide (caustic soda). This is irrespective of the casting process, be it sand or investment casting⁽⁵⁾. Silica cores used for steel castings are removed with caustic soda, either as aqueous solution by a refluxing process⁽⁶⁾. This process however is not suitable for aluminium and its alloys that are attacked by sodium hydroxide. It is therefore desirable to find an alternative material combination for casting these alloys. At present, there is no ideal ceramic core system for such light alloys⁽⁶⁾.

The project will seek to use established water-based core technology to produce core shapes, but will seek to modify the composition such that the core can be removed from inside aluminium and alloy castings using a much less aggressive leaching agent than caustic soda, for example dilute acetic acids. An additional environmental benefit of the use of dilute acids is the alleviation of some of the problems of waste disposal. Commercial quantities of caustic soda cannot be flushed away, whereas weak acids pose less of a problem.

Process requirements limit alternative candidate materials to be used in conjunction with existing plaster and fused silica. Additional components will be added to determine the optimum core composition for dimensional stability, strength and other mechanical properties, but most importantly those, which will allow easy removal with a more environmentally friendly reagent. The ultimate suitability as a casting core will also be evaluated.

2.0 LITERATURE SEARCH

2.1 CORES

A core is a ceramic or metal shape inserted in a mould to form the interior part of a casting that cannot be shaped by the pattern. A core could be a simple round cylinder form needed to core a hole through a hub of a wheel or bushing or it could be a very complex form used to create the water cooling channels in a cast engine block and the outside cylinders. Casting processes are unique in their ability to incorporate ⁽⁷⁾ internal cavities or re-entrant sections with relative ease. To produce these features, however, it is often necessary to use cores as part of the mould.

A core consists of two parts. These are the main body and the core prints. There can be one or more core prints. The prints are essential to support the core in the mould and provide a venting route for the escape of gases.

Cores can be made of metal, plaster, investment and ceramic materials as well as core sand. Cores should be collapsible or readily removable after casting whilst retaining sufficient strength at high temperature to withstand the hydrostatic forces of the molten metal and early thermal contraction of the metal as it solidifies.

2.1.1 CORE BOXES

Core boxes are an important part in core making or moulding processes. The core boxes may be made of either wood or metal. Wood is a more popular material since it is easy to glue or otherwise join, freely available and of low cost. Metal core boxes are preferred where cores are to be mass-produced ⁽⁵⁾ or a more durable containment is required. Metal core boxes may be made using cast iron, steel, brass, bronze or

aluminium alloys. Core boxes used to produce ceramic moulded cores are the same as those used for normal sand core. The quality of the finished core can be no better than the core box it is produced from and this criterion will apply to the types of core boxes used. Ceramic slurries tend to react with the surface paints ⁽⁵⁾ often used on wood core boxes. It is usually necessary to completely strip and refinish with binder compatible coatings.

The cavity in the core box must be of the exact shape and size of the core required, allowing for a known shrinkage. Difficulty may be encountered while assembling the core if core prints and cavity are not of accurate dimensions. Simple geometry cores can be produced in a 2-part split mould, whilst more complex ceramic cores will be injection moulded into a complex multi-part metal die. Some cores may be produced in more than one piece. Such split cores are assembled before the casting process. Most ceramic cores may be produced using “loose piece” core moulds. Multi cavity core moulds can produce typically four different pieces at a time, which can be used individually or assembled to produce a single casting.

2.2 CORE CLASSIFICATION

There are many ways in which cores can be classified depending on their type of shape, positioning, material and casting technique. Depending on the type of the casting process, the core can be either disposable or permanent. For example, in die-casting the cores used may be permanent while in sand casting the cores are usually disposable. There are occasions where either type can be used, such as in gravity die-casting. Positioning refers to the altitude, whether the core is placed on a horizontal or vertical axis. The different types of cores are described in the succeeding sections.

2.2.1 SAND CORES

Sand cores are made of dry silica sand mixed with a binder. The dry silica sand is shaped and clay-free. The binder cements the sand together until it is cured. Sand core mixes can be mixed in a muller or paddle type mixer or in small amounts on the bench by hand. Cores are made by ramming the sand into the core box, placing the core on a core plate⁽⁸⁾. The core box cavity is previously dusted with parting powder. Figure 1 shows a complex assembled multi-part sand core for a hydraulic control block.



Figure 1. Example of a complex sand core⁽⁹⁾

The properties of the sand in a core depend on ingredients of the mix, operations of mixing, core making, and baking. Proper mixing is performed so that binder is uniformly distributed around the particles. Sand core density is an important factor related to collapsibility and strength. Sand cores rammed to high density resist collapse and display greater hot strength. They may suffer from trapped gases however, which may result in hot tearing in a casting.

Sand cores can be further classified as green or dry sand cores. The former are made from the same sand as the rest of the mould⁽⁷⁾. The disadvantage of green sand cores is their low strength in comparison with the dry sand cores that are cured at elevated temperatures. Sand cores have major difficulty with precision and surface finish.

Since the ceramic cores have higher accuracy, the sand cores were removed from commercial sand foundries. The introduction of ceramic cores has benefited ⁽⁵⁾ sand foundries with smooth precision internal cavities at competitive costs and reduced time.

2.2.2 CERAMIC CORES

The accuracy and surface finish that commercial casting industries demand on cast parts is not easily achievable from sand cores. The successful development of ceramic cores offered accuracy and surface finish on cast internal cavities at competitive costs and impressive lead times ⁽¹¹⁾. Ceramics including plaster are major materials used in core making. Additional inorganic materials were added to improve the core strength, setting time and other properties. These are considered in more detail in the next section. Figure 2 shows an example of different ceramic core shapes.



Figure 2. Examples of different ceramic core shapes ⁽¹⁰⁾

There are two methods of making ceramic cores, preformed and poured (moulded) cores. Preformed cores are made by extrusion or injecting a slurry (core mix) with pressure into a specially made die. The poured cores are produced from slurries (core mix) prepared and poured into core boxes. The advantages and disadvantages are described in Table 1.

CORES	ADVANTAGES	DISADVANTAGES
PREFORMED	<ul style="list-style-type: none"> - High precision castings - Thin and finely detailed section - high strength 	<ul style="list-style-type: none"> - Higher tooling cost - Highly specialised technique - Wastage and rejection - limits type of alloys used
POURED	<ul style="list-style-type: none"> - Mass production castings - Diluted acids in removal - All alloys can be used 	<ul style="list-style-type: none"> - Relatively low strength - Fast setting ceramic slurry - Take longer time to remove

Table 1. Advantages/disadvantages of preformed and poured cores

The use of casting ceramic cores is known in certain applications, which require the cores to satisfy a number of characteristics and strict quality criteria such as resistance to high temperatures, absence of reactivity, dimensional stability and good mechanical characteristics. Among the applications that impose such demands are aeronautical applications, such as casting of turbine blades for turbojet engines. The improvement of casting processes, having evolved from equi-axed casting to casting with directional or monocrystalline solidification and in particular with the process known as "lost wax casting" or investment casting, has increased still further demands made on core properties. These fields of application are connected with precision casting processes. In all cases, the use of a core is involved in the manufacture of hollow parts.

2.2.3 PREFORMED CORES

The manufacture of preformed cores is a highly specialised process, requiring substantial capital equipment and technical investment. The core making process may be divided into 6 steps.

1. Die construction – metal, plaster or wood dies
2. Material mixing – time, formulation, test
3. Injection or slip pour – time, temperature, pressure, rate
4. Assembly - (if necessary)
5. Firing – arrangement, time, temperature
6. Inspection - dimensions, removing parting line.

The die usually is a metal (hardened tool steel), also aiding heat transfer if a hot mix is used. The raw materials consist of an aqueous mixture of fine ceramic powder with inorganic materials and binders. A vacuum may be applied to remove the air from the core mixture.. These materials may be warm or cold when mixed and to remove air from the mixture a vacuum is often applied. An enclosed impeller core box, for example, will usually have a narrow central opening for slurry entry.

The mix slurries are prepared by carefully mixing the aggregate and binder, avoiding excessive turbulence. Temperatures of the formulated raw materials, injection pressure, rate of injection ⁽¹²⁾ and time of injection are all controlled in the process. The mix materials are injected or poured into the die under controlled conditions. Vacuum centrifugal equipment has been adopted that allows pressurised centrifugal moulding of slurries in an enclosed vacuum. The cores are then removed from the mould and may be supported on ceramic “slumpers” to protect them from

deformation in the “green” state. Controlled and often complex firing cycles are then needed to retain control of shrinkage and dimensional accuracy. Figure 3 shows a typical ceramic preformed core in the green state.



Figure 3. Preformed core for an racing car exhaust component⁽¹⁰⁾

Preformed cores are also used in the production of Formula 1 motor racing cooling systems, oil feeds, turbo inlet ducts, brake ducts, exhausts, side inlets, wing mirrors, fire bottles⁽¹⁰⁾.

2.2.4 SLIP POURED CORES

Poured (moulded) ceramic cores are produced from a quick setting slurry (described as a core mix). The core slurry is prepared by careful mixing of ceramic powders, e.g. fused silica and binders. Setting times of the slurry are usually adjusted to typically about four minutes. A core box will usually have a narrow central opening for slurry entry. The core-box is located and locked inside the moulding machine.

As conventional venting is not possible, there may be a risk of entrapped air within the core-box. This tends to give a lower density to an upper core surface resulting in very poor finish^(11, 12) and twist out of shape in curing. Centrifugal means of filling the core box can be employed to minimize air entrapment. Poured cores are also used

in hand rails, handles, catch tanks, water header tanks, hollow reinforcing for hatches , cycle frames and parts, canoe paddles and many others. Figure 4 shows an external pump impeller core being inserted into the cope and drag portions of a ceramic mould.



Figure 4. Core being inserted into drag after curing⁽¹¹⁾

2.3 CORE DRYING

Core drying may be performed on supporting plates or core carriers or “slumpers”. Core plates are made of cast aluminium that has been machined to a flat surface. A core plate may be used in sand core storing and for the baking process. Compositions plates can also be used to carry and bake⁽⁸⁾ the cores. These plates are made from suitably heat resisting materials.

Batch and continuous type core drying furnaces may be used depending on the kind of core built and production requirements. Portable racks may be used in batch furnaces, for example, drawer type racking in which kind the cores are placed. A large core might take days to dry or a small core might take just a few hours. The drying time is controlled by the rate of travel on the conveyor for continuous type processes. Generally, all core-drying furnaces are air-circulated type, which distribute the heat

uniformly. It also carries away the moisture saturated ⁽⁷⁾ air from the cores. Figure 5 shows a conveyor core-drying oven used to dry the ceramic cores.

Green ceramic cores can exhibit distortion from stresses induced in the core from the moulding and ambient cooling operations. It can exhibit dimensional variations from one core to the next in a production run of the green ceramic cores.



Figure 5. Conveyor core drying furnace ⁽¹³⁾

2.3.1 FIRING OF CORES

Other than metal cores, all cores may be given a controlled “pre-heat” to reduce the thermal shock of firing that may have a very adverse effect on core properties. The green ceramic core is subjected to a flaming treatment ⁽¹⁴⁾ where the core is impinged with an alcohol flame to increase core strength before being subjected to a higher temperature firing. This process will also remove any residual moisture content on cores. The time and amount of firing of the core depend entirely on the composition of the core, the type of binder used and the size of core. The binder changes chemically and molecularly from liquid to solid by oxygen absorption and polymerisation ⁽¹⁵⁾.

Suitable firing is very important for the good performance of the core. If a core is heat treated for a small amount of time, it could give off much gas during pouring and solidification. A core heated for a long period may become brittle and collapse under the force of the molten metal or again during solidification. Heating of the ceramic core with the flexible weight bags⁽¹⁴⁾ confirms the core to a surface of the “setter” (placing the green ceramic core) to reduce distortion of the core and improve yields of cores within preselected dimensional tolerances.

A ceramic core includes several ceramic layers. The ceramic core controls the shrinkage rate of the outer ceramic layers⁽¹⁶⁾ during firing allowing higher density on the outer layers. Ceramic materials after firing are characterized by an interconnected network of porosity and solid phase or particles of ceramic within a porous matrix. The density of the fired compact varies with the particle size of the materials employed. For 1 to 50 μm the density of the fired compact is from about 40 to 75 %⁽¹⁷⁾.

2.3.2 CORE FINISHING AND INSPECTION

A number of operations may be performed on heat-treated cores before they are set in the mould. Core finishing and inspection are required operations adopted to ensure a quality product and to prevent defective cores getting into castings. These may include core cleaning; a dimensional check may be performed. A variety of gauges (measuring devices) may be used in core inspection processes⁽¹⁸⁾ before they are sent to the moulding floor or core storage, e.g. X-ray diffraction, surface finish, dimensional gauge. These operations may be performed after the core “pre-heating” (removing excess moisture and increasing the strength of green ceramic core). If the

processes are adopted in the core pre-heating process it may add extra time and cost. The cores should be smooth, free of flash or projections, to be ready for the core-setting operation⁽¹¹⁾.

2.3.3 CORE ASSEMBLY

Some cores are of one piece and may be set directly into the mould after cleaning and sizing. Other cores are assembled from two or more pieces before they can be used. Core assemblies may be held together by pasting and bolting.

Adhesives are most commonly used on small cores. Usually a proprietary mixture of talc, dextrin, flour, molasses, water, or other ingredients, is applied to the surfaces to be joined. The paste may put on by means of a paste bulb, brush, or by dipping with a pasting fixture. A pasting fixture consists of a metal rack, which is lowered into the tank of paste and is then raised against the prepositioned cores⁽¹⁸⁾, depositing paste at the desired points. Care must be exercised to avoid sealing off vent holes and grooves when cores are pasted. The pasted surfaces are pressed together and allowed to dry so that a strong assembly results. It is often desirable to mud the joint lines to prevent metal penetration and give a smoother casting surface. Pasted and mudded cores are then dried to avoid the danger of blow defects in the castings.

Cores may bolt together if pasting does not produce a sufficiently strong assembly. Bolting is useful in larger core work. Recessed holes are left in the cores so that they may bolt together. The bolt and nut heads are covered over with a core plug or mud. Leading similarly requires mating male and female features in the core parts to join. These are bonded with molten lead. When the lead freezes, it holds the core together,

just as a bolt. These processes are applied in manufacturing gas turbine engines (e.g. turbine blade or vane castings). A multi-wall assembly is made by coating first a thin wall ceramic core with wax plastic, a second similar ceramic core using temporary locating pins, holes are their drilled through the ceramic core⁽¹⁹⁾. A locating rod is inserted into each drilled hole and the second core is then coated with wax or plastic.

This core assembly procedure is quite complex, time consuming and costly because of use of the multiple connecting and other rods and drilled holes in the cores to receive the rods. In addition, this core assembly procedure can result in a loss of dimensional accuracy and repeatability of the core assemblies.

2.3.4 CORE SETTING

Core setting is the process of placing cores in moulds. The cores must be accurate in size and positioned accurately with respect to the mould cavity. Cores are positioned in the mould by core prints. When metal is poured, cores may rise unless they are securely anchored. Advance planning is required so that the cores will be correctly positioned and firmly held when metal is poured.

Small cores are placed in the mould by hand. Larger cores may require a hoist or crane service. A number of cores may be assembled and set in the mould at a time. This usually requires an assembly fixture e.g. for automotive motor-block cores. When transferred to the mould, the core assembly may be bolted together or held together by the fixture. Fixtures or gauges are needed when numbers of cores are assembled at one time since dimensional error may otherwise occur.

2.4 CORE RAW MATERIALS

Cores are made using sand, metal, plastic, inorganic materials, composites, and ceramics. The core raw materials are chosen depending on core shape, casting alloy, and other parameters. Different types of core making process using sand and plaster were discussed in Sections 2.2. This section will describe widely used core raw materials used for this project.

2.4.1 FUSED SILICA

Fused silica or vitreous silica is a non-crystalline form of silicon dioxide (SiO_2), having a high degree of atomic structural randomness (characteristic of a liquid). It is manufactured by the carbon arc fusion or melting of high purity glass sand at temperatures above 1710 °C.

Fused silica occurs in quartz and cristobalite. Quartz and cristobalite can be inter converted when heated. These processes are slow because the breaking and re-forming of bonds is required. The rates of conversion are profoundly affected by the presence of impurities. Standard cristobalite is prepared by firing fused silica to 1550 °C for twenty-four hours. It is then cooled, lightly ground and heated for another twenty-four hours, with intermediate “grinding”, to produce cristobalite with relatively high purity. The evidence show that cristobalite in fused silica ⁽²⁰⁾ could induce a compressive stress due to the abrupt volume increase at elevated temperatures.

The rate of crystallization of fused silica to cristobalite can depend on several parameters (e.g. type of impurity, impurity content, etc). The cristobalite content increases when fused silica ceramic cores are fired at more than 1300 °C. The fused

silica phase change results in change in dimension, shrinkage and reaction with other elements. Dimensional control of the silica core is excellent since cristobalite exhibits very little densification. Microstructural ⁽²³⁾ examination reveals that, in some cases, commercial ceramic core compositions employ very large particles (>100 μm). The addition of large particles may serve to lower shrinkage and mechanical strength.

Slow cooling of molten SiO_2 or heating any solid form to the softening temperature gives an amorphous material that is glassy in appearance. Fused silica can be obtained as various grades from millimetre to sub-micron. Fused silica is more than 99.7% pure and has reduced cristobalite levels, typically produced by the Minco Company. Table 2 Shows the fused silica and other elements present. The typical chemical composition of fused silica (both grain and flour sizes) is given.

ELEMENTS	SiO₂	Al₂O₃	Fe₂O₃	TiO₂	K₂O	CaO	Na₂O
Volume %	>99.7 %	<1,700 ppm	<280 ppm	<250 ppm	<120 ppm	<75 ppm	<50 ppm

Table 2. Fused silica compositions produced by Minco ⁽²¹⁾

Fused silica based ceramic cores are often used in investment casting and superalloy gas turbine components. Fused silica ceramic cores contract and change in length when subjected to more than 1300 °C ⁽²²⁾. However once fired at higher temperatures, fused silica has a relatively low, constant, apparent coefficient of thermal expansion between 500° and 1100 °C. The ceramic cores made by fused silica are leached out using aqueous solutions of sodium hydroxide. Table 3 shows the properties of fused silica.

State	White powder	Melting point	1800 °C
Density	2.201 g/cm ³	Co.Ther.Expansion (200°-700 °C)	0.5 -0.6 × 10 ⁻⁶ / °C.

Table 3. Properties of Fused silica⁽⁷⁷⁾

2.4.2 MAGNESIUM OXIDE

Magnesium Oxide (MgO) is formed commercially by heating magnesite between 600° and 1000 °C, which drives off most of the CO₂. It has good thermal conductivity and electrical resistance at high temperatures. The applications include high temperature refractories, abrasives, special cements and a variety of other metallurgical applications. Magnesium readily forms bonds to carbon because Mg has a high “polarizing ability” and a decided tendency for its compounds to have non-ionic behaviour⁽²⁴⁾. The thermal co-efficient of expansion is $13.5 \times 10^{-6}/^{\circ}\text{C}$ at 100 °C.

A water-soluble magnesium oxide core was trialed for castings in superalloys. Magnesium oxide and calcium phosphates⁽⁶⁸⁾ cores were used to cast superalloys in an effort to avoid the use of silica based cores, these being difficult to leach using a non-corrosive leaching medium. The magnesium cores were releasing gases, brittle and difficult to use when making castings of superalloys. It was also found that halogen could react undesirably with the superalloy during casting. A 75% magnesium oxide and 25% magnesium chloride⁽⁶⁹⁾ mixture was used to form a slurry. After pouring into the required shape, it was fired to >1700 °C.

Using magnesium oxide makes the mixed slurry⁽⁶⁴⁾ set very quickly. The magnesium oxide is also used as a mould material. The mould material consists of 50 to 80 parts per 100 parts of magnesium oxide, other materials and colloidal silica. Once mixed

with colloidal silica, it forms a ceramic slurry that can readily set in hot water ⁽⁶⁴⁾ within four to ten minutes. The set ceramic casting is dry heated to 100 °C for one to two hours. It is also claimed that adding high percentage of magnesium oxide leads to quick setting of the ceramic cast. For current research purposes, magnesium oxide was purchased from the Sigma Aldrich Company. Table 4 shows the properties of Magnesium oxide.

State	powder form	Melting point	2800 °C
Density	3.52 g/cm ³	Solubility in water	Insoluble

Table 4. Properties of Magnesium oxide ⁽⁵⁹⁾

2.4.3 LITHIUM CARBONATE

Lithium Carbonate (Li₂CO₃) is a fine white powder, less soluble in hot than cold water. It is generally stable when exposed to the atmosphere. Lithium carbonate reacts easily with strong acids and is frequently used for the manufacture of other lithium salts and suitable for use in most glass, ceramic ⁽²⁵⁾ and chemical applications. It is poorly soluble in water: only 1.33 grams dissolve in 100 cm³ at room temperature. Its solubility decreases at higher temperatures. The isolation of lithium from aqueous extracts of its ores capitalizes on this low solubility.

The apparent solubility of lithium carbonate increases tenfold under a mild pressure of carbon dioxide. For this current research, it is used as an accelerator to increase the hardness of the core. Lithium carbonate is an important industrial chemical. It forms low-melting point fluxes with silica and other ⁽⁸²⁾ materials. Glasses derived from lithium carbonate are useful in ovenware. Cement sets more rapidly when prepared with lithium carbonate.

Most of the ceramic cores have accelerators. This is either to increase the setting time or to add extra strength with filling materials. Chalk and limestone are some of the additives used in the production of ceramic cores. This increases the setting time and supports additional bonding with filler material. The additions of several additives or accelerators⁽⁸³⁾ have been claimed to increase the setting time and strength of cores. Table 5 shows the properties of lithium carbonate,

State	White in colour	Melting Point	618 °C
Density	2.11g/cm ³	Solubility in water	13.3 g dm ⁻³

Table 5. Properties of Lithium carbonate⁽²⁵⁾

2.4.4 CALCIUM SILICATE

Calcium silicate is an insulator due to its low thermal conductivity at elevated temperatures. It has a low bulk density and high physical water absorption. It is used as an anti-caking agent and an antacid. It has excellent creep resistance, is insoluble in water and has good mechanical properties in relation to weight. It is environmentally friendly; it can be formed in any shape easily and can be re-useable. It has low thermal capacity and a low thermal shrinkage property⁽²⁶⁾. For current research purpose, calcium silicate was purchased from Sigma Aldrich Company.

State	White or cream colour	Melting Point	1500 °C
Density	1.82 g/cm ³	Solubility in Water	insoluble

Table 6. Properties of Calcium silicate⁽²⁵⁾

Calcium silicate is used in ceramic cores to break the bonds after casting is performed. It is claimed that 45 % (the weight ratio of CaSiO₃) in a ceramic core can be leached using water. With calcium silicate, sodium chloride, potassium⁽⁷²⁾ chloride

and sodium metasilicate is added to produce a core. These materials have higher melting temperature than aluminium.

2.4.5 PLASTER

Calcium sulphate hemi-hydrate($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) is obtained through the partial or total dehydration of gypsum at a temperature ranging from 120 to 400 °C. The structure and properties of the final product are directly dependent on the chosen calcination condition such as temperature and pressure ⁽²⁷⁾. Depending upon the chosen calcinations, plaster is divided into two types, alpha and beta plasters.

Casting applications use alpha plaster formulations for its high mechanical strength. Beta plasters are used in certain ceramics applications and construction. If a greater concentration of water is used, the mix will be more fluid, the strength reduced and the setting time lengthened. With less water, the converse applies. The major differences are in terms of setting time, strength and workability. A number of these are detailed below,

DENTAL PLASTER is a formulated hemi-hydrate plaster produced from naturally occurring gypsum mineral. It is used in dentistry during the “flasking” operation in the production of dentures. It can be mixed with dental stone plaster for the production of model bases. It has a setting time of around 11 minutes.

FINE CASTING PLASTER (FCP) is a beta calcium sulphate hemihydrate plaster produced from naturally occurring gypsum mineral. Beta plaster crystals have a high specific surface and high water demands. Beta plaster casts have high porosity, low

mechanical properties and are used in lightweight building applications or moulds in ceramic applications for their absorbent properties. It has a setting time of 12 to 30 minutes.

CRYSTICAL R(CRP) is an alpha- high strength hemihydrate plaster produced from high purity gypsum mineral. Alpha type plaster is used mainly in industrial plaster formulations for its high mechanical strength. This plaster type is a compact crystal with a low specific surface and low water demands to produce hard, low porosity casts. It has a setting time of 15 minutes.

CRYSTICAL LX(CRX) is an alpha calcium sulphate hemihydrates, off-white plaster produced from high purity gypsum mineral. It is designed for use where high strength, surface hardness, and dimensional accuracy are required. This plaster is exceptionally strong and durable, has negligible expansion on setting and gives an excellent surface finish when cast. It has a setting time of 60 minutes⁽²⁷⁾. For current research purpose, plaster is purchased by BpB formula. Table 7 shows the properties of different type of plaster used for current research purpose.

PLASTER	MOR(2 h -dry)	Expansion(2 h)	Density(dry)
DENTAL	6.4 MN/m ²	0.35%	850 kg/m ³
FCP	12 MN/m ²	0.3%	750 kg/m ³
CRP	44.8 MN/m ²	0.55%	1670 kg/m ³
CRX	36 MN/m ²	0.08%	950 kg/m ³

Table 7. Properties of different type of plaster⁽²⁷⁾

2.4.6 TRI-SODIUM CITRATE ($C_6H_5Na_3O_7$)

Its known as citric acid tri-sodium salt. It occurs as two forms, crystal and anhydrous. It is odourless and occurs as colourless crystals or as a white powder. It is very much soluble in water (70 g/100 g of water). Applications are in pH buffering (keeping constant pH when exposed to strong acids), as an anticoagulant agent ⁽²⁸⁾, flavour enhancer and modifier. Tri-sodium citrate dehydrate is used to make cores. The increased MgO percentage had the mixed slurry setting quickly. For research process, it is used to reduce the setting time of a mixed slurry.

Tri-sodium citrate has very limited application in ceramic core production. This is due to its high cost. Tri-sodium citrate is also used as an accelerator. It helps in the core removal process by reacting with acids to form salts, which are soluble in water.

2.4.7 CALCIUM CARBONATE ($CaCO_3$)

Calcium carbonate occurs naturally, mainly in two crystalline forms. The calcite form is colourless and occurs as marble, limestone and chalk. Aragonite is the second crystalline form and is metastable with respect to calcite. It is found in coral shells. The calcium carbonates are only sparingly soluble in water and on strong heating decompose to give the oxide and carbon dioxide.

It reacts with all strong acids (e.g. HCl), releasing carbon dioxide on heating above 840 °C to form ⁽²⁹⁾ calcium oxide. The main use of calcium carbonate is in the construction industry. It is widely used as an extender in paints, filler in plastics, adhesives, sealants, and decorating fillers. For research purpose, it has been tried out as a replacement for fused silica (filler material).

2.4.8 BINDERS

Binders serve to hold the grains together and impart strength, resistance to erosion and to breakage, and degree of collapsibility. They may be classed as organic, inorganic, and metallo-organic binders. Organic binders are combustible, and destroyed by heat. They contribute a degree of collapsibility to the core-sand mixture. Inorganic binders are not combustible and may have considerable strength at high temperatures, resistance to erosion, and may be relatively non-collapsible depending on their nature.

Colloidal silica (e.g. Ludox AM[®]) consists of discrete uniform spheres of silica that have no internal surface area or detectable crystallinity. It is an amorphous silicon dioxide. During drying, the hydroxyl groups on the surface of the particles condense by loss of water resulting in the formation of siloxane bonds (Si-O-Si), coalescence and inter-bonding⁽³⁰⁾. These are effective binders for all types of granular and fibrous materials, especially where use at elevated temperature is required.

Various other types of binders are known, both organic and inorganic, for adhering the core material particles together. Linseed oil-based organic binders are widely used; they contain a resin and thinner, to provide good⁽⁹⁵⁾ wetting and workability properties. Other known organic binders, including plastics of the urea and phenol-formaldehyde groups, are also widely used. These organic binders generally entail a two-part polymer resin which is set during the core forming process so as to form an extremely durable core.

Inorganic binders (like colloidal silica) are widely used in ceramic cores. The binders are used to get higher strength, strong bonding, smooth surface finish and smooth

flow slurry mix. The “greencast” process is a new lost-core technology for the production of hollow parts from thermoplastics and thermosets⁽⁷³⁾ using water-soluble cores. The core moulding material consists of an inorganic binder. Cores must be coated with a material that is made especially for these cores. The cores as well as the coating are inorganic, ecologically safe, water soluble and recyclable. Table 8 shows the properties of Ludox[®] AM,

State	fluid	Melting Point	0 °C
Density(20 °C)	1.2 g/cm ³	Solubility in Water	70%

Table 8. Properties of Ludox[®] AM colloidal silica⁽³⁰⁾

WATER is not usually thought of as a binder. Unless an optimum percentage of water exists in the core mixture, binders and additive do not function properly. Failure to use enough water results in not developing green strength from “cereals” and poor scratch hardness, low tensile strength after firing.

Excessive water causes stratification⁽³¹⁾ of binders by seepage, stickiness, surface crusting and causes other core making and drawing problems.

Water has some very unusual properties. It has higher density than ice and its melting point is abnormally high. Water reacts with potassium, sodium and calcium liberating hydrogen. The water molecule contains lone pairs of electrons and is extensively polarized. This factor makes the water molecule a strong proton⁽⁶¹⁾ acceptor. This is the reason why strong acids have a more vigorous reaction with water. Water is a very strong solvent, dissolving many types of substances. Water also has high surface tension properties (filling voids and pores).

2.4.9 ACIDS AND BASES

Acids are substances that can donate hydrogen ions to bases⁽⁷⁴⁾ (proton acceptors). Inorganic acids dissolve in water, giving a solution with a hydrogen ion activity greater than in pure water. Acids are corrosive, react with metal and bases. Acids are divided into two different types, strong and weak acids. Nitric, hydrochloric, sulphuric and hydrofluoric acids are considered as strong acids. Citric and acetic acids are weaker acids. The weak acids have lower concentration of ions relating to lower reaction with metal and bases.

Strong and weak acids is used to leach out ceramic cores from castings. The degree of dilution depends on the ceramic core material, shape, size and application. For any ferrous metals, strong acids and caustic alkalis are used to remove the ceramic cores. For light metals and superalloys, diluted nitric acid and bases such as potassium⁽⁷⁰⁾ hydroxide are used. It is proposed to use various acids during the experimental stage of the work. The basic properties are described below

ACETIC ACID (CH₃COOH). Acetic acid is a clear, colourless liquid with a pungent odour. Its systematic name is ethanoic acid. It will be referred to as acetic acid in the thesis. It is soluble in water and most organic solvents e.g. alcohol or glycol. Pure, water-free acetic acid is a colourless liquid that attracts water from the environment (hygroscopy), and freezes below 16.7 °C to a colourless crystalline solid. It is considered a weak acid because at standard temperature and pressure the dissociated acid exists in equilibrium with the un-dissociated⁽⁸⁴⁾ form in aqueous solutions, in contrast to strong acids, which are fully dissociated. The product may

react violently with amines, other bases, strong oxidising agents, and perchloric acid.

Table 9 shows the properties of acetic acid.

State	Liquid or crystals	Melting Point	16.5 °C
Viscosity	1.22 cP at 25 °C	Boiling point	118 °C
Molar Mass	60.05 g/mol	Solubility in Water	Fully miscible

Table 9. Properties of Acetic acid ⁽⁸⁵⁾

Acetic acid is corrosive to many metals including iron, magnesium, and zinc, forming hydrogen gas and metal salts called acetates (ethanoates). Aluminium, when exposed to oxygen, forms a thin layer of aluminium oxide on its surface which is relatively resistant, so that aluminium tanks can be used to transport acetic acid.

NITRIC ACID (HNO₃) It reacts with water or steam to produce heat and toxic, corrosive, and flammable vapours depending on the amount ⁽³³⁾ of nitrogen dioxide present. Nitric acid reacts violently with alcohol, turpentine, charcoal, sodium and potassium compounds, and organic refuse. It reacts explosively with metallic powders and many reducing agents, attacking all base metals except aluminium and special chromium steels. It is soluble in cold and hot water.

Nitric acid is a colorless, yellow, or red liquid. It is used in the manufacturing of pharmaceuticals, rubber chemicals, inorganic and organic nitrates, and organic chemicals. Nitric acid causes a characteristic choking. As a corrosive liquid it will attack some forms of plastics, rubber, and coatings. It is not combustible, but may give off poisonous oxides of nitrogen and an acid fume when heated. Table 10 shows the properties of nitric acid.

State	Colourless Liquid	Melting Point	-42 °C
Viscosity	2.6 cP	Boiling point	83 °C
Molar Mass	63.01 g/mol	Solubility in Water	miscible

Table 10. Properties of Nitric acid ⁽⁸⁵⁾

CITRIC ACID (C₆H₈O₇) shares the properties of other carboxylic acids. When heated above 175 °C, it decomposes through the loss of carbon dioxide and water. At room temperature, citric acid is a white crystalline powder. It can exist either in an anhydrous (water-free) form or as a monohydrate. The anhydrous form crystallizes from hot water, whereas the monohydrate forms when citric acid is crystallized from cold water. The monohydrate can be converted to the anhydrous form by heating it above 74 °C. Citric acid also dissolves ⁽³²⁾ in absolute (anhydrous) ethanol (76 parts of citric acid per 100 parts of ethanol). Citric acid is used extensively in the food, beverage, cosmetic, and pharmaceutical industries as well as being a very important ingredient for use in natural body care and cosmetic recipes. Table 11 shows the properties of citric acid.

State	White solid	Melting Point	153 °C
Viscosity	2.6 cP	Boiling point	decomposes
Molar Mass	192.027 g/mol	Solubility in water	133 g/100cm ³

Table 11. Properties of Citric acid ⁽⁸⁵⁾

SODIUM HYDROXIDE (NAOH) Caustic soda forms a strong alkaline solution when dissolved in a solvent such as water. It is deliquescent and readily absorbs carbon dioxide from the air; it should be stored in an airtight container. It is very soluble in water with liberation ⁽⁸⁶⁾ of heat. It also dissolves in ethanol and methanol, though it exhibits lower solubility in these solvents than potassium hydroxide. It is

insoluble in ether and other non-polar solvents. Sodium hydroxide does not attack iron or copper, but other metals such as aluminium, zinc and titanium are attacked rapidly. Sodium hydroxide reacts readily with carboxylic acids to form their salts and is even a strong enough base to form salts with phenols. It is used in many industries, mostly as a strong chemical base in the manufacture of pulp and paper, textiles, soaps and detergents and as a drain cleaner. Table 12 shows the properties of NaOH,

State	White solid	Melting Point	318 °C
Viscosity	4.0 cP	Boiling point	1390 °C
Molar Mass	39.991 g/mol	Solubility in Water	111 g/100cm ³

Table 12. Properties of Sodium Hydroxide ⁽⁸⁵⁾

POTASSIUM HYDROXIDE (KOH) dissolution in water is strongly exothermic, producing substantial amounts of energy in form of heat, sometimes increasing the temperature up to boiling point. Concentrated aqueous solutions ⁽⁸⁷⁾ of potassium hydroxide are called potassium “lyes”. It is a highly basic compound, forming strongly alkaline solutions in water and other polar solvents, capable of deprotonating many acids, even weak ones, and decomposing some inorganic as well as organic materials. It reacts violently with acid and is corrosive in moist air toward metals such as zinc, tin, lead forming a combustible, explosive gas. It rapidly absorbs carbon dioxide and water from air. Contact with moisture or water will generate heat. It is used in chemical manufacturing including potassium carbonate and other potassium chemicals. Table 13 shows the properties of potassium hydroxide,

State	White solid	Melting Point	360 °C
Viscosity	0.005 cP at 20 °C	Boiling point	1320 °C
Molar Mass	56.10 g/mol	Solubility in Water	1100 gdm ³

Table 13. Properties of Potassium Hydroxide ⁽⁸⁵⁾

2.5 CERAMIC CORE PROPERTIES AND CORE TESTING

2.5.1 CERAMIC CORE PROPERTIES

The desirable properties of a ceramic core are easy to list but far more difficult to attain perfection in practice. Controlled strength, surface finish, refractoriness, ease of removal and consistency are some of the desirable properties. A trade-off in core properties may be required in order to produce accurate dimensions in the casting.

The core should have sufficient strength to avoid breakage during the casting process but not be too strong as to restrain solidifying metal that may lead to hot tearing or cracking of the casting. Ceramic cores must be stable in air or vacuum throughout the temperature cycle. Ceramic cores need to resist the effects of, or reaction with, other process materials. The core may be subjected to temperatures above 800 °C in the casting process. Poor refractoriness (heat resistance) would lead to core softening, resulting in unacceptable dimensional control of the casting.

2.5.2 THREE POINT BENT TEST

Modulus of Rupture (MOR) test results represent the relative relationships between the load bearing capabilities of a ceramic core and its area. The MOR number is the inherent strength of the ceramic. The hot MOR test was not done in this research due to non-availability of equipment and financial constraints in outsourcing the work.

The stress-strain behaviour of brittle ceramics is not usually ascertained by a tensile test for three reasons. It is difficult to prepare and test specimens having the required geometry. It is difficult to grip brittle materials without fracturing them. Ceramics fail after only about 0.1% strain. This necessitates that tensile specimens are perfectly

aligned in order to avoid the presence of bending stresses. A more suitable transverse bending test is most frequently employed, in which a specimen having either a circular or rectangular cross section is bent until fracture using a three point loading technique⁽³⁴⁾. At the point of loading, the top surface of the specimen placed in a state of compression, whereas the bottom surface is in tension. Figure 6 shows the three point loading scheme.

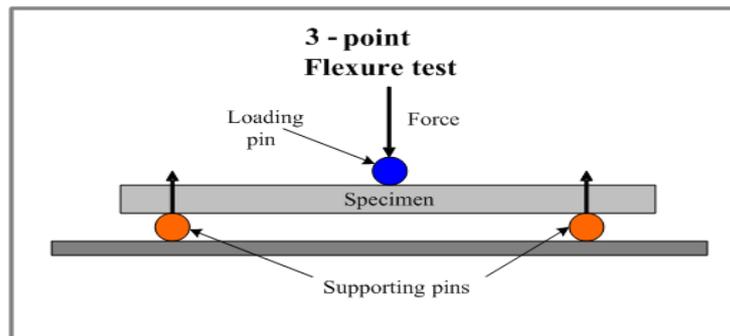


Figure 6. The three point loading scheme⁽³⁵⁾

Stress is computed from the specimen thickness, the bending moment, and the moment of inertia of the cross section. The maximum tensile stress exists at the bottom specimen surface directly below the point of load application. The stress at fracture using the flexure test is known as the modulus of rupture (MOR). When the cross-section is circular, the equation to calculate MOR is developed below:-

The magnitude of the direct stress, σ , is related to the bending moment, M , by the general equation,

$$\frac{\sigma}{L} = \frac{M}{I} = \frac{E}{R} \dots\dots\dots \text{Equation 1}$$

where, $I = \pi \frac{R^4}{4}$ is the second moment of area of the section, E is Young's Modulus,

R is Radius of curvature of the neutral axis at the section.

$$M = \frac{FL}{4} * R * I = \pi \frac{R^4}{4}$$

$$\sigma = \frac{M}{I} L$$

The equation for calculating a modulus of rupture for a circular section is,

$$\sigma_{fs} = \frac{F_f L}{\pi R^3} \dots\dots\dots \text{Equation 2}$$

where 'F_f' is the load at fracture, 'L' is the distance between support points and 'R' is specimen radius.

2.5.3 WEIBULL ANALYSIS

The presence of undefined flaws in this type of material makes prediction of the probability of failure difficult. Since failure occurs at the largest favourably orientated flaw, if the stress to cause fracture is distributed over a range of values, then we define fracture for any particular stress level (Weibull).

The Swedish engineer, Weibull ⁽³⁸⁾ first formulated the distribution of strength of brittle materials; he defined a probability of survival F_S. The Weibull distribution is given by the expression,

$$F_w = 1 - \exp\left\{\left(\frac{x - \mu}{\sigma}\right)^m\right\} \dots\dots\dots \text{Equation 3}$$

where 'x' is strength or fatigue cycles, 'σ' is a position parameter (1-1/e), μ is a lower strength boundary below which no specimen fails and m is a width parameter (Weibull modulus).

With increasing specimen volume (under stress), there is an increase in the probability of flaw severity and consequently flexural strengths will exist over a range. The Weibull distribution can be of great use because it is possible to extrapolate back to the very low probability of failure region and to select a design stress to give a desired failure rate. The Weibull modulus becomes more important than the position parameter (or average strength) when the reliability of the least reliable results is required.

The distribution overview plot from Minitab[®] has four different functions. The probability density function (pdf), Weibull (Shape and Scale), Survival function and Hazard function.

Probability density function (pdf) displays the shape of the chosen distribution and provides the relative chance that the product will fail in any particular interval of time.

It is defined relating to Weibull probability density function(pdf).

$$f(x) = \frac{\beta x^{\beta-1} e^{-\left(\frac{x}{\alpha}\right)^\beta}}{\alpha^\beta}, x > 0 \dots\dots\dots \text{Equation 4}$$

where β is Weibull shape and α is scale parameter.

Weibull or Probability plot is used to assess how well the chosen distribution fits the data. If the points hug the fitted line, then it is reasonable to use that distribution to model the data. The points on the plot are the estimated percentiles based on a “nonparametric” method. The line is based on the fitted distribution.

X-axis $\ln(\text{failure data})$ and Y-axis $\ln(-\ln(1-p))$

Survival function plot depicts the probability that the core will survive until a particular time. The survival plot displays the reliability of the core over time. The Y-axis displays the probability of survival and the X-axis displays the reliability measurement (failure data). The survival function $S(t)$, also known as reliability, function $R(t)$, represents the probability a unit survives beyond time.

$$R(t) = 1 - F(t) \dots \dots \dots \text{Equation 5}$$

where $F(t)$ is the pdf for the chosen distribution

Hazard function provides the likelihood of failure as a function of how long a unit has lasted (the instantaneous failure rate at a particular time). The hazard plot shows the trend in the failure rate over time. It is given by

$$h(t) = \frac{f(t)}{1 - F(t)} \dots \dots \dots \text{Equation 6}$$

where $f(t)$ and $F(t)$ are the probability density function(pdf) and cumulative density function(cdf) respectively of the chosen distribution.

Weibull analysis⁽³⁶⁾ is a method for modelling data sets containing values greater than zero, such as failure data. The analysis can make predictions on survival probability and compare the reliability of competing product designs. For this reason, the Weibull approach is frequently employed with ceramics.

The Weibull distribution is defined by shape (β) and scale (α). The Weibull shape parameter (also known as Weibull modulus), β , indicates whether the failure rate is increasing, constant or decreasing. A value of $\beta < 1.0$ indicates that the product has a decreasing failure rate⁽³⁷⁾. This scenario is typical of "infant mortality" and indicates that the product is failing during its "burn-in" period. A value of $\beta = 1.0$ indicates a

constant failure rate. Frequently, components that have survived burn-in will subsequently exhibit a constant failure rate. A value of $\beta > 1.0$ indicates an increasing failure rate. The Weibull characteristic life, called α , is a measure of the scale, or spread, in the distribution of data. The plotting procedure in Minitab[®] is in section 3.4.2.

2.5.4 APPLICATION OF WEIBULL ANALYSIS

The lower the Weibull modulus(m), the greater the variability of strength. For ordinary chalk, m is about 5, and the variability is great. Brick, pottery, and cement are similar to chalk. The engineering ceramics (e.g. SiC, Al₂O₂) have the values of Weibull modulus of about 10, the strength varies rather less. The Weibull modulus for steel is about 100, though showing some variation in strength⁽³⁹⁾, but it is small; such a material can be treated as having a single, well, defined failure stress.

Weibull analyses are also applied in casting techniques. For pressure die-castings, Weibull modulus is often between 1 and 10, whereas for much gravity die-casting it is between 10 and 30. For good quality aerospace castings a value between 50 and 100 is more usual.

2.5.5 Y, K_{IC} and 'a' DEFINITION AND EQUATION

The principal drawback of ceramic materials is a disposition to catastrophic fracture in a brittle manner with very little energy absorption. The crystalline and noncrystalline ceramics usually fracture at room temperature to an applied load. The crystalline ceramics usually have a crack growth through the grains and along specific crystallographic planes.

Such crack growth can lead to brittle fracture that is calculated from stress intensity factor (K). There are different ways of defining and calculating fracture toughness. The ceramic materials calculation adopts plain strain fracture toughness (K_{IC}). The initial fracture toughness (K_{IC}) is defined by,

$$K_{IC} = Y(a/W)\sigma\sqrt{\pi a} \dots\dots\dots \text{Equation 7}$$

where ‘ σ ’ is the Modulus of Rupture of cracked specimen, ‘Y’ is stress intensity factor or as a function of both cracks length (a) and component width (W) i.e. as ‘Y (a/W)’.

For example, for a plate of infinite width having a through-thickness crack ‘Y (a/W)’ is 1.0, while for a plate of semi-infinite width ⁽⁶⁷⁾ containing single centre or edge crack of a length, Y (a/W) is 1.1. Mathematical expressions for ‘Y (a/W)’ in terms of a/W are required for components of finite dimensions.

To calculate the ‘Y’ for a circular section with a semi-circular notch on three points bending a closed form solution was developed ⁽⁷⁹⁾. This compares well with the current semi-circular notch on the ceramic cores subjected to three point bending. The equation is given by:

$$Y\left(\frac{a}{D}\right) = 0.6257 - 0.5856\left(\frac{a}{D}\right) + 2.0824\left(\frac{a}{D}\right)^2 \dots\dots\text{Equation 8}$$

where ‘a’ is crack length and ‘D’ is diameter of the solid bar.

For relatively thin specimens, the value of K_C will decrease with increasing specimen thickness or diameter. Eventually, K_C becomes independent of thickness or diameter,

at which time the condition of plane strain is said to exist. Experimentally, it has been verified that for plane strain conditions,

$$Diameter \geq 2.5 \left(\frac{K_{IC}}{\sigma} \right)^2 \dots\dots \text{Equation 9}$$

The constant value of K_C for thicker specimens is known as the plane strain fracture toughness (K_{IC}). This definition can be mathematically expressed by the following expression:

$$K_{IC} = \sigma Y \sqrt{\pi a} \dots\dots\dots \text{Equation 10}$$

In Equation 8 , ‘Y’(a/D) is stress intensity factor or a dimensionless factor dependent on the test geometry of the crack and material dimensions, ‘σ’ is the Modulus of Rupture of fracture ceramic core; ‘a’ is the length of surface crack or half-length of a central crack.

The plane strain fracture toughness of material is defined as the material’s ability to resist brittle fracture during the presence of a crack or flaw. The plain strain fracture toughness is a quantitative way of expressing a material's resistance to brittle fracture when a crack presents. If a material has a large value of fracture toughness it will probably undergo ductile fracture. Brittle fracture is very characteristic of materials with a low fracture toughness value.

Ceramic materials have a much lower K_{IC} value than metals. The low K_{IC} value reflects the fact that ceramic materials⁽³⁹⁾ are very susceptible to cracks and undergo brittle fracture, whereas metals undergo ductile fracture. The K_{IC} values for ceramic materials are smaller than for metals, typically smaller⁽⁶⁷⁾ than $10 \text{ MN } \sqrt{\text{m}}$. Due to

the brittle nature of ceramics, the standard test methods, to give load/extension graphs, cannot be used for the reasons described above. Three point bend tests are therefore used. To determine the maximum critical defect size ‘ a_c ’, the MOR and K_{IC} are kept constant. Then the maximum critical defect size, from re-arrangement of Equation 7, is

$$a_c = \frac{1}{\pi} \left(\frac{K_{IC}}{Y\sigma} \right)^2 \dots \text{Equation 11}$$

where ‘ σ ’ is the MOR of non-cracked specimen.

The derivation of critical defect size is to avoid the occurrence of catastrophic failure. The examining of structural components for defect size leads to understanding of the reliability. Several non-destructive test techniques have been developed that permit detection and measurement of both internal and surface defects.

2.5.6 K_{IC} IN CERAMICS

Brittle ceramics and glasses typically break following elastic deformation, without plastic deformation. The plain strain fracture toughness is strongly affected in glass-infiltrated alumina reinforced ceramics with minimal variations⁽⁴¹⁾ of the grain size, shape and orientation. Conversely, in the case of pressable ceramics (such as lithium disilicate) this has no effect on K_{IC} . Table 14 shows the fracture toughness for some of the ceramic materials.

Materials	Alumina	Magnesia	Limestone
K_{IC} (\sqrt{MN})	3-5	3	0.9

Table 14. K_{IC} for ceramic materials⁽⁴¹⁾

2.5.7 THERMAL SHOCK ON CERAMICS

The inherent brittleness of ceramics combined with their common use at high temperatures make thermal shock a major concern. Fracture caused by sudden changes in temperature is a problem with ceramics. Some ceramics (e.g. glass) will only take a thermal “shock” of 80 °C before they break, others (e.g. silicon nitride) will stand change of 500 °C. It may be measured by dropping a piece of the ceramic, heated to progressively higher temperatures, into cold water. The maximum temperature drop, which the ceramic survives, is a measure of its thermal shock resistance. High performance engineering ceramics (e.g. silica, zircon), have a small coefficient of expansion ⁽⁴²⁾ which indicate they can survive thermal shocks without fracturing.

2.6 CASTING ALUMINIUM AND ITS ALLOYS

2.6.1 CASTING ALLOYS

ALUMINIUM and its alloys are classified as either cast or wrought using a four-digit number that indicates the composition of the various alloys. The principal alloying elements include copper, magnesium, silicon, manganese, and zinc. The primary material for aluminium production, aluminium oxide, is produced from bauxite. Aluminium does not occur in metallic form in nature but in its compounds. The chief limitation of aluminium is its low melting temperature (~660 °C), which restricts the maximum temperature at which it can be used. Aluminium and its alloys are characterized by a relatively low density, high electrical and thermal conductivity and a resistance to corrosion in most common environments. The mechanical strength of aluminium may be enhanced by subsequent cold work and alloying ⁽⁴³⁾.

A typical and commonly used aluminium casting alloy is LM 25 conforming to BS1490:1988. The resistance to corrosive attack in seawater and a marine atmosphere is high, hence its application in this type of environment, where moderate strength is required. It offers better machinability and mechanical properties than many other aluminium alloys. The physical and chemical properties of casting Al-LM25 are shown in Table 15 and 16.

Coefficient of Thermal Expansion (per °C @ 20-100°C)	0.000022
Thermal conductivity (cal/cm ² /cm/°C @ 25 °C)	0.36
Electrical conductivity (% Copper standard @ 20 °C)	39
Density (g/cm ³)	2.68
Melting range (°C) approx.	615-550

Table 15. Physical properties of Al- LM25⁽⁴⁴⁾

The chemical composition of LM25 is

Element	Cu	Mg	Si	Fe	Mn	Ni	Zi	Le	Sn	Ti	Others	Al
%	0.2	0.6	6-7.5	0.5	0.3	0.1	0.1	0.1	0.1	0.2	0.05	Remainder

Table 16. Chemical composition of Al-LM25⁽⁴⁴⁾

MAGNESIUM weighs about two-thirds as much as aluminium, and one-fourth as much as iron. Mechanical properties are similar to those of aluminium. The corrosion resistance of magnesium is low, especially to marine atmospheres; it must be surface treated and painted where weight is important, in such articles as portable saws, lawnmower housings, and automobile engine components. Melting is often considered a drawback and has to be carried out under a protective atmosphere. The surface of molten magnesium must be protected from the atmosphere to prevent oxidation and the formation of “dross” on the surface of the metal. The most

commonly used magnesium alloy has a chemical composition including aluminium, zinc, manganese, copper, silicon, iron and nickel⁽⁴⁵⁾.

COPPER and its alloys were among the earliest metallic objects made from molten metal. Since copper was found as native metal, it has been worked into artifacts. The main bulk of copper production comes from ores, which are primarily mined for their copper content. The principal alloying elements include nickel, zinc, and combinations of different metals. The melting point of copper and its alloys with gold, tin, and zinc are low enough to be within the range of temperatures, which can be reached by wood and charcoal fires⁽⁴⁶⁾. Sand casting accounts for the greatest percentage of castings produced in copper and its alloys. Copper alloy die-casting can be accomplished only with a relatively few alloys. Copper is much more limited in casting in terms of shape and size due to its high density.

Copper casting alloys are specified for services where superior corrosion resistance, electrical conductivity, good bearing surface qualities, and other special properties are desired. The tolerance for impurities is normally greater in castings than in their wrought copper because of the adverse effect on hot or cold workability⁽⁴⁷⁾.

2.6.2 CASTING METHODS

Gravity Die Casting. Gravity or permanent die-casting is a process where the molten metal is poured into metal moulds or dies without any external pressure. The moulds are made in two or more parts that fit together easily and can be clamped during the actual casting process. They are shaped so as to permit the casting to be readily extracted. Castings can be manufactured by operation of dies manually or by

automatic devices or through die casting machines depending on the quantity of production.

The dies are made typically using grey cast iron and occasionally steels. Casting die design is important in considerations where undercuts and hollow shapes are produced with the help of cores. It is even more important in such circumstances to provide adequate vents and risers; the wall thickness of the mould must be carefully considered in relation to the cross-sectional area of the castings. This may cause prevention of local solidification or inadequate feeding of other parts of the casting. Indeed, limitations of size and complexity are the main disadvantages. Dies are generally designed using 3-D solid modelling software, (discussed in Experimental Section).

Sand or Plaster of Paris cores may be employed for production of interior details. The mechanical design of such permanent moulds can be very complex depending on the precise usage and shape of casting required so that each case must be considered individually. The maximum height of pouring aluminium metal into a mould is 13 mm⁽⁵⁴⁾ because large areas of oxide films will be entrained in the melt. The gravity die casting process is suitable for high volume production of non-ferrous alloy castings of aluminium, magnesium, and copper and zinc base alloys and to limited extent for cast iron castings.

Gravity die casting is capable of achieving typically 20% better mechanical properties than of sand casting since a faster rate of solidification imparts a finer grain size⁽⁴⁸⁾.

The process can be automated. Table 17 shows the comparison of different casting processes.

		Sand Casting	Gravity Die Casting	Pressure die Casting
1	Tolerance (Min)	± 0.75 mm	± 0.5 mm	± 0.1 mm
2	Surface Finish	12 to 24 microns	4 to 12 microns	1 to 2 microns
3	Maximum Weight (Al alloy)	No limit	300 Kgs	10 Kgs
4	Max size (area) Al Alloy	No limit	800 x 500 mm	500 x 500 mm
5	Minimum wall thickness (Al Alloy)	3.5 mm	3.0 mm	1.0 mm
6	Production Quantity (EOQ)*	100	2000	100,000
7	Minimum Cored Hole size	10 mm	6 mm	2 mm
8	Machining allowance (min)	2.5 mm	1.5 mm	0.5 mm
9	Mechanical Properties (Scale)	1	1.25	1

*EOQ: Economical order quantity.

Table 17. Comparisons of different aluminium casting processes⁽⁴⁸⁾

Pressure Die Casting, the fastest of all casting processes, ranks and competes with stamping, die forging, plastic injection moulding where large numbers of identical parts are required. The molten metal is forced into the mould cavity under high pressure. The hot (metal flows into chamber) and cold chambers are the two type of machine principles used in pressure die-casting. The metal enters the die through the gate in a high velocity jet, the dimensions and velocity of which depend upon the dimensions of the gate and runner. This jet strikes the opposite face of the die cavity and spreads out in all directions filling the die cavity rapidly and breaking down into turbulent flow as the cavity fills up. Solidification takes place under pressure. The die itself is normally machined from a steel forging, spark erosion methods being used for intricate⁽⁴⁹⁾ cavities.

Aluminium, magnesium, copper-base and other high melting alloys use cold chamber principle (metal is ladled into chamber). The use of pressure die-castings reduces the time (assembly) and expense (tooling) required producing the product ⁽⁵⁰⁾. High pressure casting results in conforming very closely to the exact shape of the mould, a high degree of dimensional accuracy can be achieved without resort to subsequent machining. Aluminium pressure die-casting cannot easily be electroplated and anodising is not very practical. The high silicon alloys are predominantly used in high-pressure aluminium die-casting. Aluminium castings range from a few grams up to 25 kg in weight.

Sand Casting is a means of producing rough metal castings using a mould made from sand formed around a replica of the object to be cast that is removed once the sand has been compacted. Medium sized “green” sand moulds are enclosed in flasks that consist of two parts, upper part (cope) and lower part (drag). The shapes of the desired casting are simulated by a pattern ⁽⁵¹⁾ around which the moulding sand is formed. The patterns are made of wood or metal, also in two parts that can be fitted together. The green sand is rammed around one-half of pattern placed in the drag. The same process is carried out in the cope with spurs (runner) and gates (risers).

After patterns are removed, the shape of the mould cavity is identical with the shape of the pattern. Before casting, the surface of the mould is dried by flaming with a torch and cope and drag are assembled together. The best practice is to fill the mould from the bottom through one or more gates allowing suitable vents to facilitate the escape of air, together with a riser that perform the dual function of venting the air

and filling metal. The low density of aluminium needs great care to ensure proper filling of the mould.

When sand casting aluminium alloy, gas content and grain refinement are the two special points that must be taken into consideration. Gas content should be controlled at a reasonably low level to avoid the formation of blowholes and porosity. De-gassing tablets (hexachlorethane) are used to control the gas content. In slowly cooled castings there may be a tendency for large crystals to form, leading to reduced mechanical properties. Addition of titanium and boron can prevent the grain refinement in aluminium alloy sand casting⁽⁵²⁾. Figure 7 shows the sand casting with sand cores.

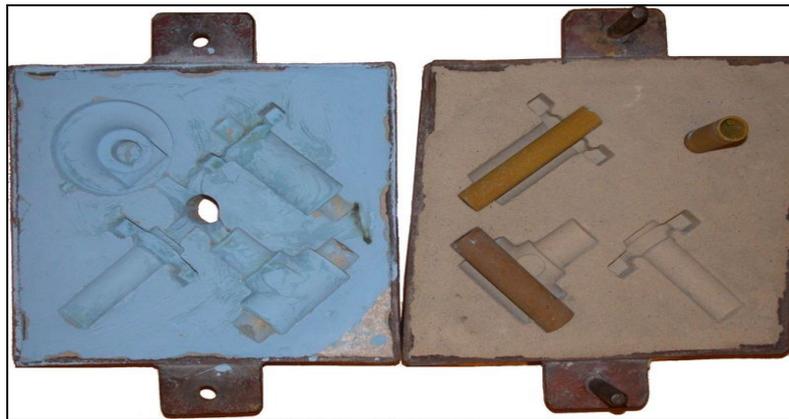


Figure 7. Sand casting with sand cores⁽⁵⁹⁾

2.6.3 CASTINGS DEFECTS

The form and position of defects in castings is often of greater importance than the size. A large pore in a low stressed area of the casting may be far less detrimental than a small “bifilm” in a sharp corner subject to a high tensile stress⁽⁵³⁾. Fins or flashing can occur if a die is worn or not clamped tight. It may also result in run-outs. Cast part

surfaces may have “pinholes” caused by the absorption of hydrogen or carbon monoxide when the moisture content of sand is high or the metal poured is not sufficiently degasified.

Uncontrolled solidification of the metal causes shrinkage in cast part. Shrinkage may occur due to inadequate filling in riser or feeder. Surface turbulence will produce porosity, cracks, and unreliability in leak tightness and mechanical properties. Cold shut defects are usually caused by metal at low temperature poured too slowly. Mis-run defects occur when a portion of the castings fails to run due to cold metal, slow pouring, insufficient hydrostatic pressure (non-fluid due to badly gassed or oxidized metal).

2.6.4 CORE CASTING DEFECTS

When molten metal is poured on a core, the heated core composition and binder may cause large volumes of gas to be generated in the core. Well-designed cores are designed so that the gas can escape through the core prints and so be lost from the mould.

If the venting of the core is poor or the metal is poured too slowly, the pressure of gas in the cores may rise to a level higher than the pressure of molten metal, with the result that a bubble is forced out into the solidifying metal. Large bubbles formed in this way are initially large but may fragment such that the number is increased ⁽⁵⁴⁾.

Figure 8(a) shows schematically a core blow-trapped bubble evolved after initial solidification and 8(b) an exfoliated dross defect produced by gas from a core blow prior to any solidification.

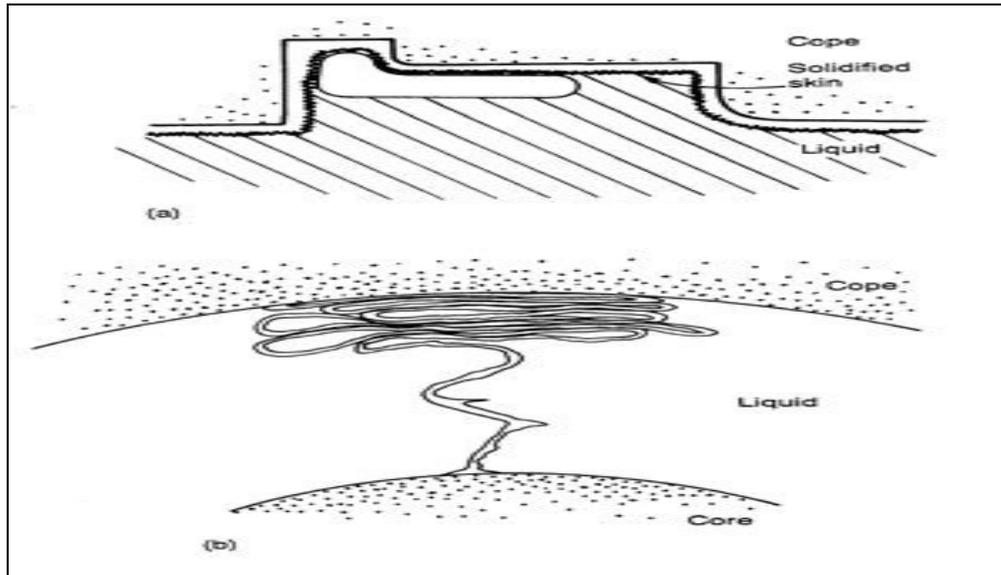


Figure 8. (a) Core blow (b) Defect caused by gas from cores before solidification

(54)

Blows can also form from moulds. The volumes of gas that are generated inside the mould have to be considered. The higher metal pressure effectively suppresses any bubbling of gas through the core. The best solution to the evolution of gases from cores is the use of a binder for the core that suffers little or no evolution of gas.

The cores can be the source of casting defects. Poor or defective cores should not be delivered to the moulding operations. Cores may still be the source of casting defects even after providing a clean, good, dimensionally accurate core. The sand core mixture, the method of locating, and securing cores in the mould can cause defects. Thermal effects can cause the core to burn out and momentum of the molten metal can cause it to shift from where it was assembled.

In the gravity filled running systems, to reduce bubbles in castings requires offset stepped basins or other advanced filling systems. The requirement also demands

properly engineered ⁽⁵⁴⁾ and manufactured “sprues”. Loose sand may result in dirt defects in castings. Core raise defect occurs when the core floats and moves up close to the “cope”. A weak, undersize, or improperly assembled core causes this defect. Core shift defect can cause a thinner wall on one side of casting and thicker on the other side.

Metal penetration defects occur in large castings where the core becomes heated up to the melting point of the metal before it freezes. Soft ramming of mould with a low sintering point seems to aggravate metal penetration defect. A hard core, which does not allow the casting to contract may rupture or tear the metal. This will give rise to hot tear defects, which are very common in non-ferrous castings.

2.7 CERAMIC CORE REMOVAL

Simple sand cores are the easiest to remove from the casting, since the binders are degraded by the high temperatures. In larger castings, with larger open cavity cores, the method of hydro-blasting can be used. Other core removal process involves the use of chemicals and different non-destructive technology.

In the past, the ceramic core has been removed from the cast turbine airfoil by an autoclave technique, open kettle or other technique. One autoclave technique involves immersing the casting in an aqueous caustic solution (e.g. 45% KOH) at elevated pressure and elevated temperature (e.g. 1.72 MN/m² and 177 °C) ⁽⁸⁸⁾ for an appropriate time to dissolve or leach the core from the casting. An exemplary open kettle technique ⁽⁸⁹⁾ involves immersing the casting in a similar aqueous caustic

solution at ambient pressure and elevated temperature (e.g. 132 °C) with agitation of the solution for a time to dissolve or leach the core from the casting.

In 1974, the British Cast Iron Research Association (BCIRA) reported loosening CO₂ core sand in the internal passage of heavily cored castings⁽⁵⁵⁾ using “lithotripsy”. A Hungarian steel foundry similarly experimenting with electro-hydraulic sand removal used a spark-gap electrode discharging system in water to create a pressure wave, which impacted the casting and the core. The core was disrupted by both direct impact of the pressure wave on the core prints and the pressure transmitted through the casting walls. The latter phenomenon made use of the natural vibration characteristics of the casting to transmit tensile and shear forces to the core.

There have been other attempts to use mechanical energy to remove cores involving ultrasonic vibration. Initial trials were made to use ultrasonic vibration alone to remove cores from turbine blades. This evolved into the use of ultrasonic waves⁽⁵⁶⁾ generated and propagated through hot caustic alkalis in which the castings were immersed, to combine mechanical and chemical core removal techniques. Although the development of the use of ultrasonic vibration was considered successful, the process never became established as a production process.

2.7.1 SHOCK WAVE CORE REMOVAL

Electro-hydraulic shock waves can be used to break up the core mechanically, so that it falls out of the casting. Shock waves, which are generated in a fluid such as water by the instantaneous discharge of an electric current, have sufficient energy to fracture

ceramic materials. This energy dissipates as the pressure waves spread out from the electrodes.

Electrical energy, generated from a large capacitor bank, can also be focused. The intensity of the pressure waves transmitted to the part from the shock wave in the water depends on the acoustic impedance of the ceramic core itself, so it is necessary to know the strength of the materials used in ceramic cores⁽⁵⁷⁾.

Wave speed, stress wave propagation and compressive strength are calculated to ensure core removal will not fracture or distort the castings. The stresses calculated may be high enough to cause re-crystallization in single crystal superalloy castings.

2.7.2 WATER JET CORE REMOVAL

Water jet removal processes may also be used to clear the passages of core materials. Since the entire core removal process is carried out under water, there is substantially reduced risk of dust pollution in the work environment. The effectiveness of water removal technology also depends on the mechanical properties of the core material and the geometry of the casting.

A stream of water is focused on the core under great pressure and the core is progressively washed out. This procedure takes place in a purpose built facility or blasting room. “Cerabite” is an example of a typical commercial process⁽⁵⁸⁾. The process uses focused waves in a recirculation water bath for removing ceramic core. Core materials are typically removed from superalloy, aluminium and steel.

A method of removing ceramic core material from an internal passage of a super alloy airfoil casting comprising discharging pressurised fluid comprising water from a spray nozzle at an opening of the passage at an exterior root surface of the casting. This is discharging pressurised fluid is controlled ⁽⁹⁰⁾ by computer numerical control (CNC) machine.

3.0 EXPERIMENTAL TECHNIQUES

In order to achieve the aims of the project, combinations of different ceramic materials at different concentrations were used to make the cores. These materials were readily available from commercial sources. Different trials using different proportions of inorganic materials were progressed, to determine the effectiveness of the dilute acids on the leaching behaviour, subject to a material being produced which had adequate mechanical strength and could withstand the conditions encountered by exposure to liquid metal. Experimental design techniques were evaluated, e.g. Taguchi ⁽⁹⁶⁾ but were not ultimately employed. Whilst such methods may have been useful for a single outcome improvement, the multiple outcomes sought in this investigation may have been problematic. Furthermore, the interaction between the materials is complex and the use of extreme levels limits the possible outcomes.

3.1 CORE COMPOSITIONS

From an initial definition of trials, iterative core composition followed, varying different parts of the trial composition for mechanical properties, casting and core removal. Trials were undertaken at each core composition to build-up a comprehensive understanding of the effect of the compositional changes. In some core compositions, the core trials were unchanged but the grain size or particle size distribution was amended.

Core compositions [1]-[7] correspond to changes in main core trial materials and [8]-[9] involve adding replacement material from main core trial. Trials A observed the varying percentage of plaster, Trials B observed varying percentage of binder, Trials C observed the varying percentage of magnesium oxide, Trials D observed the

addition of calcium silicate and Trials E observed the change in plaster type. All core trials were formed to find the right core for aluminium and light alloys. The following tables' core compositions give a brief explanation on materials used.

- **Core composition [1]** Initial start of core trails were tried out with very coarse and medium coarse fused silica to find bonding system. This is a standard table which was reviewed for further stages.
- **Core composition [2]** Medium coarse fused silica was replaced with fine fused silica due high density. The magnesium oxide percentage was reduced to quick slurry working life.
- **Core composition [3]** Fine fused silica was used to produce core due to high density of coarse fused silica.
- **Core composition [4]** Produced core without using any additives to find the effect on core slurry working life and setting time.
- **Core composition [5]** Added required magnesium oxide percentage to increase the slurry working life.
- **Core compositions [6]and [7]** Made cores using water and binder to see effect on core strength and materials
- **Core compositions [8] and [9]** Changed the filler material to calcium carbonate and magnesium oxide additive to tri-sodium citrate to find the alternative for filler.

Apart from the Core composition [1], each and every core composition was decided from previous limitations on producing a core for aluminium and light alloys.

3.1.1 CORE COMPOSITION [1] -STANDARD

The composition [1] of Trials A to E have made up as follows, volume percentage. It also shows that 2A and 1D composition have same volumetric percentages.

TRIAL A	SiO ₂ (-120)	SiO ₂ (30/50)	MgO	Li ₂ CO ₃	Plaster	Colloidal silica (Binder/water)
1A	44	34.9	1	0.1	20	50/50
2A	34	34.9	1	0.1	30	50/50
3A	24	34.9	1	0.1	40	50/50
4A	14	34.9	1	0.1	50	50/50

TRIAL B	SiO ₂ (-120)	SiO ₂ (30/50)	MgO	Li ₂ CO ₃	Plaster	Colloidal silica (Binder/water)
1B	34	35.9	1	0.1	30	20/80
2B	34	35.9	1	0.1	30	40/60
3B	34	35.9	1	0.1	30	60/40
4B	34	35.9	1	0.1	30	80/20

TRIAL C	SiO ₂ (-120)	SiO ₂ (30/50)	MgO	Li ₂ CO ₃	Plaster	Colloidal silica (Binder/water)
1C	35	34.9	0	0.1	30	50/50
2C	34	34.9	1	0.1	30	50/50
3C	33	34.9	2	0.1	30	50/50

TRIAL D	SiO ₂ (-120)	SiO ₂ (30/50)	CaSiO ₃	MgO	Li ₂ CO ₃	Plaster	Colloidal silica (Binder/water)
1D	34	34.9	0	1	0.1	30	50/50
2D	24	34.9	10	1	0.1	30	75/25

TRIAL E	SiO ₂ (-120)	SiO ₂ (30/50)	MgO	Li ₂ CO ₃	Plaster	Colloidal silica (Binder/water)
1E	34	34.9	1	0.1	30(FCP)	50/50
2E	34	34.9	1	0.1	30(CRP)	50/50
3E	34	34.9	1	0.1	30(CLX)	50/50

FCP	Fine casting plaster
CRP	Crystcal R
CLX	Crystcal LX

Table 18. Trials A to E of core composition [1]

3.1.2 CORE COMPOSITION [2]

Core Trials A to E have reduced magnesium oxide volume percentage and fused silica mesh size changed, in volume percentage.

TRIAL A	SiO₂ (-200)	SiO₂ (30/50)	MgO	Li₂CO₃	Plaster	Colloidal silica (Binder/water)
1A	44	35.7	0.1	0.2	20	50/50
2A*	34	35.7	0.1	0.2	30	50/50
3A	24	35.7	0.1	0.2	40	50/50
4A	14	35.7	0.1	0.2	50	50/50

TRIAL B	SiO₂ (-200)	SiO₂ (30/50)	MgO	Li₂CO₃	Plaster	Colloidal silica (Binder/water)
1B	34	35.7	0.1	0.2	30	20/80
2B	34	35.7	0.1	0.2	30	40/60
3B	34	35.7	0.1	0.2	30	60/40
4B	34	35.7	0.1	0.2	30	80/20

TRIAL C	SiO₂ (-200)	SiO₂ (30/50)	MgO	Li₂CO₃	Plaster	Colloidal silica (Binder/water)
1C	34	35.8	0	0.2	30	50/50
2C*	34	35.7	0.1	0.2	30	50/50
3C	34	35.2	0.6	0.2	30	50/50

TRIAL D	SiO₂ (-200)	SiO₂ (30/50)	CaSiO₃	MgO	Li₂CO₃	Plaster	Colloidal silica (Binder/water)
1D*	34	35.7	0	0.1	0.2	30	50/50
2D	24	35.7	10	0.1	0.2	30	75/25

TRIAL E	SiO₂ (-200)	SiO₂ (30/50)	MgO	Li₂CO₃	Plaster	Colloidal silica (Binder/water)
1E	34	35.7	0.1	0.2	30(FCP)	50/50
2E	34	35.7	0.1	0.2	30(CRP)	50/50

*- similar core compositions

Table 19. Trials A to E of core composition [2]

3.1.3 CORE COMPOSITION [3]

Core Trials A to E, fused silica mesh size changed to -120, as follows.

TRIAL A	SiO₂	SiO₂	MgO	Li₂CO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/water)
1A	44.7	35	0.1	0.2	20	50/50
2A	34.7	35	0.1	0.2	30	50/50
3A	24.7	35	0.1	0.2	40	50/50
4A	14.7	35	0.1	0.2	50	50/50

TRIAL B	SiO₂	SiO₂	MgO	Li₂CO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/water)
1B	34.7	35	0.1	0.2	30	20/80
2B	34.7	35	0.1	0.2	30	40/60
3B	34.7	35	0.1	0.2	30	60/40
4B	34.7	35	0.1	0.2	30	80/20

TRIAL C	SiO₂	SiO₂	MgO	Li₂CO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/water)
1C	34.8	35	0	0.2	30	50/50
3C	34.2	35	0.6	0.2	30	50/50

TRIAL D	SiO₂	SiO₂	CaSiO₃	MgO	Li₂CO₃	Plaster	Colloidal silica
	(-200)	(-120)					(Binder/water)
2D	24.7	35	10	0.1	0.2	30	75/25

TRIAL E	SiO₂	SiO₂	MgO	Li₂CO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/water)
1E	34.7	35	0.1	0.2	30(FCP)	50/50
2E	34.7	35	0.1	0.2	30(CRP)	50/50

Table 20. Trials A to E of core composition [3]

3.1.4 CORE COMPOSITION [4]

Core Trials A to E with no MgO and Li₂CO₃, are as follows.

TRIAL A	SiO₂ (-200)	SiO₂ (-120)	Plaster	Colloidal silica (Binder/water)
1A	45	35	20	50/50
2A	35	35	30	50/50
3A	25	35	40	50/50
4A	15	35	50	50/50

TRIAL B	SiO₂ (-200)	SiO₂ (-120)	Plaster	Colloidal silica (Binder/water)
1B	35	35	30	20/80
2B	35	35	30	40/60
3B	35	35	30	60/40
4B	35	35	30	80/20

TRIAL D	SiO₂ (-200)	SiO₂ (-120)	CaSiO₃	Plaster	Colloidal silica (Binder/water)
2D	25	35	10	30	75/25

TRIAL E	SiO₂ (-200)	SiO₂ (-120)	Plaster	Colloidal silica (Binder/water)
1E	35	35	30(FCP)	50/50
2E	35	35	30(CRP)	50/50

Table 21. Trials A to E of core composition [4]

3.1.5 CORE COMPOSITION [5]

Core Trials A to E have MgO with 20/80(binder/water ratio) concentrations changed.

TRIAL A	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Binder/water)
1A	45	34.9	0.1	20	20/80
2A	35	34.9	0.1	30	20/80
3A	25	34.9	0.1	40	20/80
4A	15	34.9	0.1	50	20/80

TRIAL D	SiO₂	SiO₂	MgO	CaSiO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/water)
2D	25	34.9	0.1	10	30	20/80

TRIAL E	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Binder/water)
1E	35	34.9	0.1	30(FCP)	20/80
2E	35	34.9	0.1	30(CRP)	20/80

Table 22. Trials A to E of core composition [5]

3.1.6 CORE COMPOSITION [6]

Core Trials A to E, producing cores are produced using only binder, as follows.

TRIAL A	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Binder)
1A	45	34.9	0.1	20	100
2A	35	34.9	0.1	30	100
3A	25	34.9	0.1	40	100
4A	15	34.9	0.1	50	100

TRIAL D	SiO₂	SiO₂	MgO	CaSiO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder)
2D	25	34.9	0.1	10	30	100

TRIAL E	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Binder)
1E	35	34.9	0.1	30	100
2E	35	34.9	0.1	30	100

Table 23. Trials A to E of core composition [6]

3.1.7 CORE COMPOSITION [7]

Core Trials A to E cores using only water are produced, as follows.

TRIAL A	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Water)
1A	45	34.9	0.1	20	100
2A	35	34.9	0.1	30	100
3A	25	34.9	0.1	40	100
4A	15	34.9	0.1	50	100

TRIAL D	SiO₂	SiO₂	MgO	CaSiO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Water)
2D	25	34.9	0.1	10	30	100

TRIAL E	SiO₂	SiO₂	MgO	Plaster	Colloidal silica
	(-200)	(-120)			(Water)
1E	35	34.9	0.1	30	100
2E	35	34.9	0.1	30	100

Table 24. Core Trials A to E of core composition [7]

3.1.8 CORE COMPOSITION [8]

In Core Trials A to E the filler material is changed to CaCO₃, as follows.

TRIAL A	CaCO₃	MgO	Plaster	Colloidal silica
				(Binder/Water)
1A	45+34.9	0.1	20	20/80
2A	35+34.9	0.1	30	20/80
3A	25+34.9	0.1	40	20/80
4A	15+34.9	0.1	50	20/80

TRIAL D	CaCO₃	MgO	CaSiO₃	Plaster	Colloidal silica
					(Binder/Water)
2D	25+34.9	0.1	10	30	20/80

TRIAL E	CaCO₃	MgO	Plaster	Colloidal silica
				(Binder/Water)
1E	35+44.9	0.1	30	20/80
2E	35+34.9	0.1	30	20/80

Table 25. Core Trials A to E of core composition [8]

3.1.9 CORE COMPOSITION [9]

Core Trials A to E are with Tri- sodium citrate, as follows.

TRIAL A	SiO₂	SiO₂	C₆H₅Na₃O₇	Plaster	Colloidal silica
	(-200)	(-120)			(Binder/Water)
1A	45	34.9	0.1	20	20/80
2A	35	34.9	0.1	30	20/80
3A	25	34.9	0.1	40	20/80
4A	15	34.9	0.1	50	20/80

TRIAL D	SiO₂	SiO₂	C₆H₅Na₃O₇	CaSiO₃	Plaster	Colloidal silica
	(-200)	(-120)				(Binder/Water)
2D	25	34.9	0.1	10	30	20/80

TRIAL E	SiO₂	SiO₂	C₆H₅Na₃O₇	Plaster	Colloidal silica
	(-200)	(-120)			(Binder/Water)
1E	35	34.9	0.1	30	20/80
2E	35	34.9	0.1	30	20/80

Table 26. Core Trials A to E of core composition [9]

3.2 CORE MAKING

3.2.1 TRIAL MEASUREMENT

Components of the core trial were weighed to an accuracy of 0.001g. Calibration of the scale (Fisher brand) was carried out every 100 readings using a supplied standard. No adjustment was necessary on any occasion. The binder and water were measured using a tube meter. All trial values were converted from volume percentage to weight in gram and binder/water in cubic centimetres (50 volume percentage=25 cm³).

3.2.2 WOODEN CORE BOX

Plain softwood was used to make the core boxes. The core box dimensions were 25 mm diameter and 160 mm long. Three coats of gloss paint were applied before use.

Petroleum jelly was used as a mould release agent. Figure 9 shows the core boxes with cores.

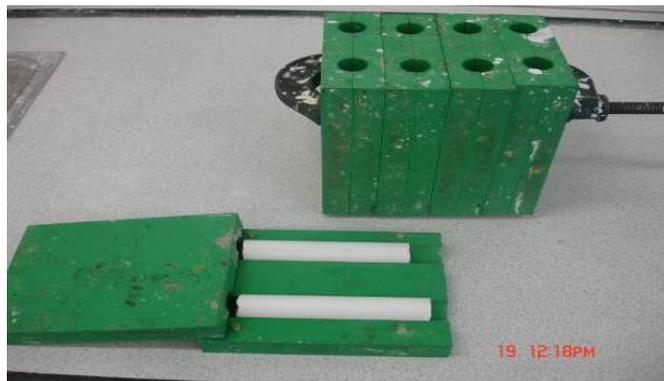


Figure 9. Core mould boxes with cores

3.2.3 MIXING, MIXED STABILITY AND POURING METHODOLOGY

Polystyrene cups were used to mix the measured core trial composition with binder and water. A hand-held mixer was used to mix the trial. Dry mixing was done for fifteen seconds. The pre-mixes of binder/water were made before the dry mixes were performed. The mixing process was continued nominally for fifteen seconds after adding binder/water. The core mix (slurry) temperature was in the range 15 to 20 °C. The stability of the mixed trials was dependent on the percentage of magnesium and plaster added to individual core trial compositions (discussed in Chapter 5). This caused certain core trials to be prepared slightly differently in comparison with nominal intentions. Air bubbles were removed from the slurry before pouring into the core box. The core box was made available as mixing was performed. The mixed core trial was poured into marked core boxes. The core boxes were left until visually dry, in order that sufficient core breakage (determined by inspection) did not occur on removal.

3.3 CORE DRYING AND FIRING

All cores were removed from core boxes after visual inspection. Room temperature drying was carried out for twenty-four hours on all cores. The cores had varying splits, voids, and visible pores on the core surface. The outer surface finishes were different for individual core compositions. Any visually surface damaged cores were removed and replaced with new cores. The dimensions of cores were measured before firing. Cores were fired at 200, 400, 600 and 800 °C in a muffle furnace for two hours. The cooling to room temperature was done at the same rate (individual temperature) inside the muffle furnace for two hours. The muffle furnaces were controlled by ElectroX™ temperature controllers. After cooling, shrinkage was evaluated from measured core dimensions. Visual inspection was made on outer surfaces to screen damage.

To measure the effect of complete moisture removal from cores, a single batch of fifteen cores were fired at constant temperature of 200, 400, 600 and 800 °C for twenty-four hours. The trial batches were measured for weight every two hours once removed from the muffle furnace, as discussed in Chapter 5.

3.4 CORE MECHANICAL PROPERTIES TESTING

Batches of ten and later twenty-five samples of each core trial (composition 1-5) were produced for mechanical property testing and one sample for casting purposes. Other core compositions (6-9) have different batch size

3.4.1 MODULUS OF RUPTURE (MOR)

To determine the flexural strength or Modulus of Rupture (MOR) of the cores, three point bend testing was used. The three-point bend test produces the maximum bending moment at the centre of the bar, at the point of contact. The test pieces were mounted in a computer controlled Lloyds LRX Universal Testing Machine (UTM) shown in Figure10.



Figure 10. LLOYD Universal test machine

A calibrated load cell of full-scale deflection of 500 N was used at a crosshead movement rate of 5 mm/minute. The span was 100 mm. All MOR testing was performed on cylindrical specimens. The diameters varied depending on the trial composition. All MOR data was recorded on the test machine computer with Lloyds Scientific software. The equations for determining the MOR are discussed in Section 2.5.2 and Equation 2.

3.4.2 WEIBULL ANALYSIS IN MINITAB

The analysis was carried out to evaluate percentage of survival probability curves on core composition [5]. The Weibull distribution parameters are defined by shape and

scale. Minitab[®] was used to calculate the Weibull parameters. It was also used to plot the survival probability of cores. Within Minitab[®], parametric distribution analysis was chosen to plot the distribution overview of core trials. The distribution overview has recorded Weibull modulus (β) also known as Shape and Weibull character (α) also known as Scale. The correlation coefficient (Corr) is also calculated. The overview plot is not relevant to current research purpose other than three parameters. The equations for determining ' β ' and ' α ' are discussed in Section 2.5.2

3.4.3 PLANE STRAIN FRACTURE TOUGHNESS TESTING

Plane strain fracture toughness (K_{IC}) tests were carried out on core composition [5]. A maximum "6 mm" semicircular notch was made on fired cores. Three point bend tests were performed on the specimens. The same computerised Lloyd LRX universal testing machine (UTM) was used to perform the tests as had been used for the MOR testing, span of 100 mm.

The fracture surface was measured to determine accurately the notch depth. Cracks were seen (manually) using a hand held cam connected to computer. It is assumed that being a ceramic material that a sharp crack exists at the root of the notch. The equations for determining the ' Y ', K_{IC} and ' a_c ' are discussed in Section 2.5.5. Figure 11 shows the diagram of fracture sample with three-point load.

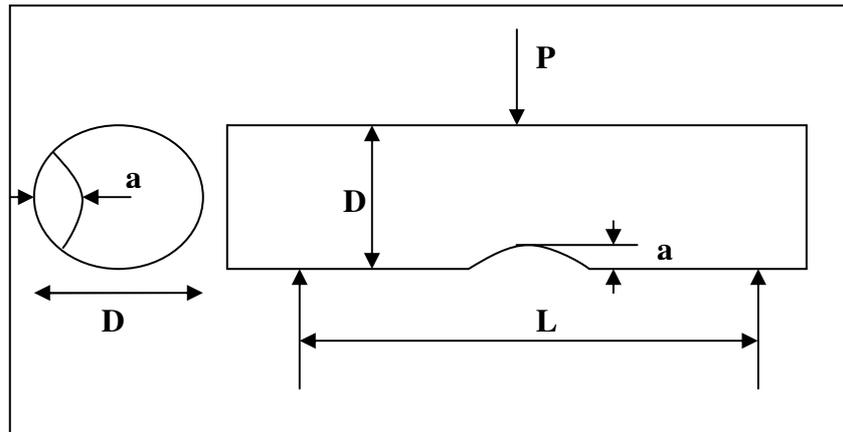


Figure 11. Fracture sample with three point bend load

3.5 CORE CASTING

Fired cores were used with a gravity die-casting process. Aluminium alloy LM25, was used for casting purposes. Core splits were first removed using silicon carbide papers. Split casting dies were designed and manufactured from cast iron. AutoCAD2003 was used to design the dies. Various machining operations were used to manufacture moulds, including turning and milling. Figure 14 shows the 2-D diagram of cast iron moulds. Since the gravity die casting is mainly used in producing aluminium, the casting system was designed to produce the dies in cast iron.

The mould cavity diameter was 26 mm by 150 mm long. Figure 12 shows the cast iron split moulds with core. An oven was used to keep moulds at a constant temperature of 250 °C prior to casting. Moulds were removed from the oven only when the casting was due to be poured. A proprietary mould release agent was applied before the cores were assembled. A G-clamp was used to hold the two halves together.



Figure 12. Cast iron split moulds with core

Before casting, polishing papers were used to remove any voids or uneven surfaces on cores. High temperature fired cores were left to cool down before casting was performed. The aluminium was melted in a gas-fired furnace at a nominal temperature of $690\text{ }^{\circ}\text{C} \pm 10\text{ }^{\circ}\text{C}$. A digital thermometer (thermocouple) was used to record temperatures. A set of six castings were poured from each melt, the castings were removed on solidification and the mould returned to the oven until a temperature of $250\text{ }^{\circ}\text{C}$ was once again attained. Figure 13 shows the pouring process to assembled mould.



Figure 13. Pouring process to assembled mould

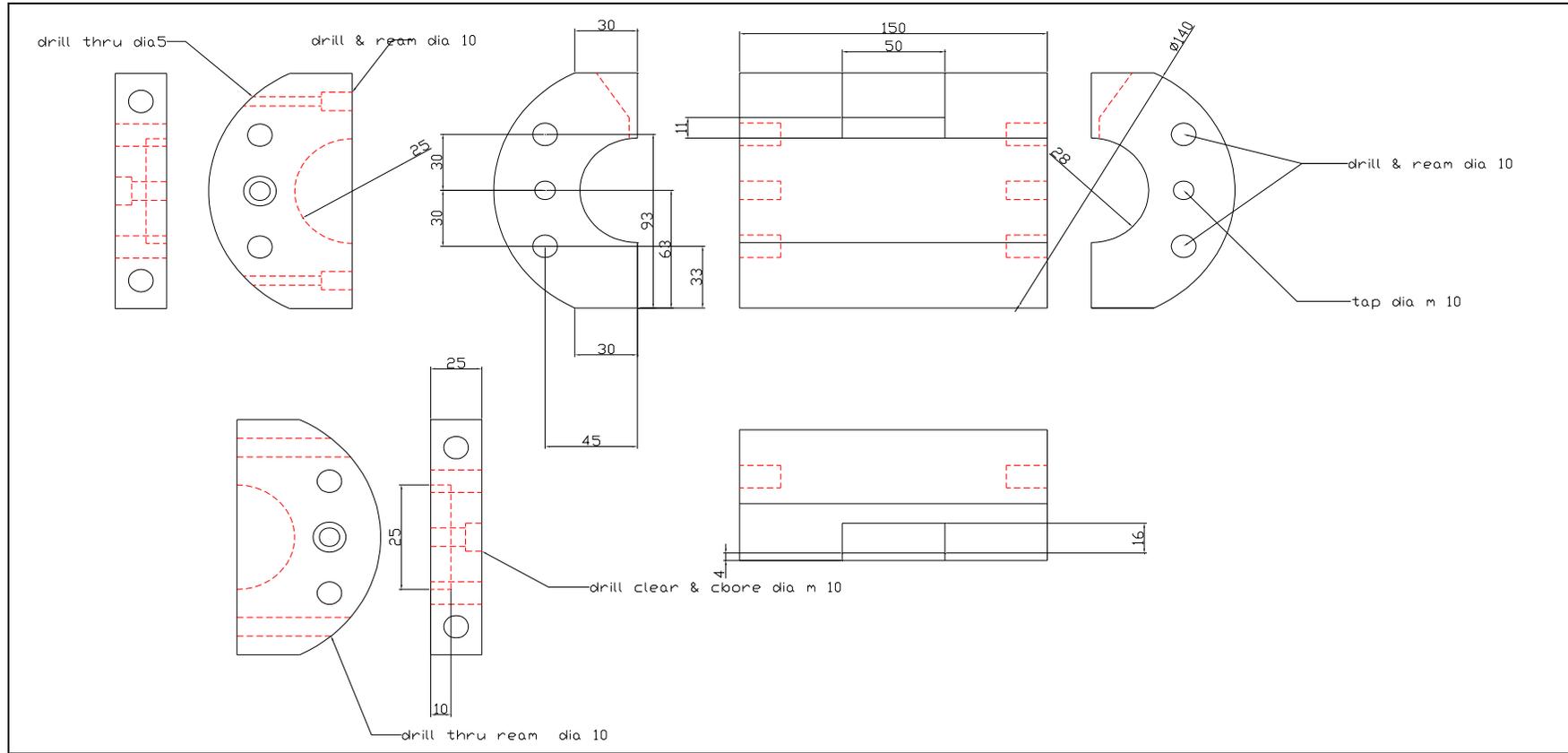


Figure 14. General arrangement of cast iron mould

3.6 CORE REMOVAL

To remove the cores from the cast part, various dilutions of nitric, citric and acetic acid and potassium hydroxide were used. Potassium hydroxide reacted with the cast part (aluminium) and its use was discontinued. Times for dissolution or softening of the core were measured and some semi-quantitative description of the ease of core removal was recorded. Dilutions of 1:1, 1:2, 1:5, 1:10, 1:50, 1:100, 1:500 and 1:1000 (acid: water) were used at the outset. Figure 15 shows the marked cast parts ready for core removal tests.



Figure 15. Marked cast parts

Checks were made every 1, 4 and 24 hours using a sharp edged iron rod. The diluted acid with the cast parts were placed in a fume cupboard. All health and safety procedures were carried out when diluting acid, dipping, removal of cast part and draining the used diluted acids. Water jet washing was used to remove cores which were obviously affected by dilute acids. Cores, which were not affected by acid, were manually removed using a drill bit and burning out aluminium. Since the application of Taguchi was not used, all different combination of dilute acid solutions were tried out.

4.0 RESULTS

The results are arranged from core manufacturing, testing, casting and removal of the core from the cast part. Evaluation of the effectiveness and suitability of the equipment and methodologies used is presented and discussed in Chapter 5.

4.1 CORE COMPOSITION [1] RESULTS

The cores of composition [1], (mixed using Ludox AM[®]/water and left to dry at room temperature) are shown typically in Figure 16.



Figure 16. Cores left to dry

The mean of slurry working life and setting time in core box with error bars are presented in Figures 17 and 18 including a calculated indication of the precision of the time measurement. The pouring was very difficult due to quick setting. This has limited the slurry flow into wooden core box leaving some settled slurry in mixed cup. Table 18 shows the 3E composition, which cannot be made (Discussed in Section 5).

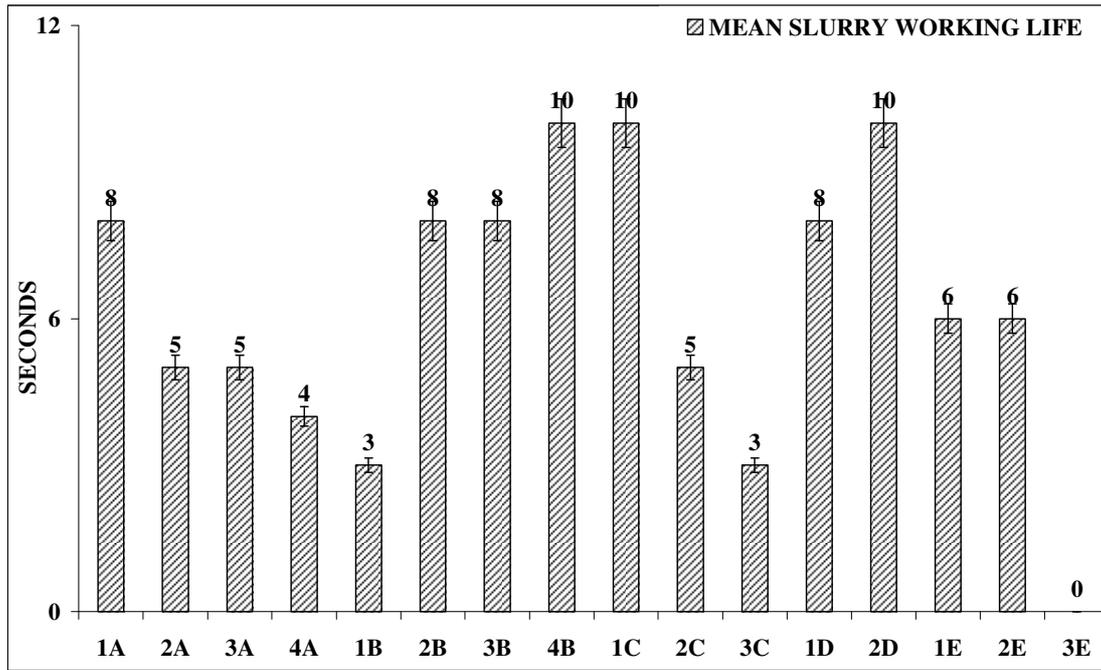


Figure 17. Mean slurry working life at core trials

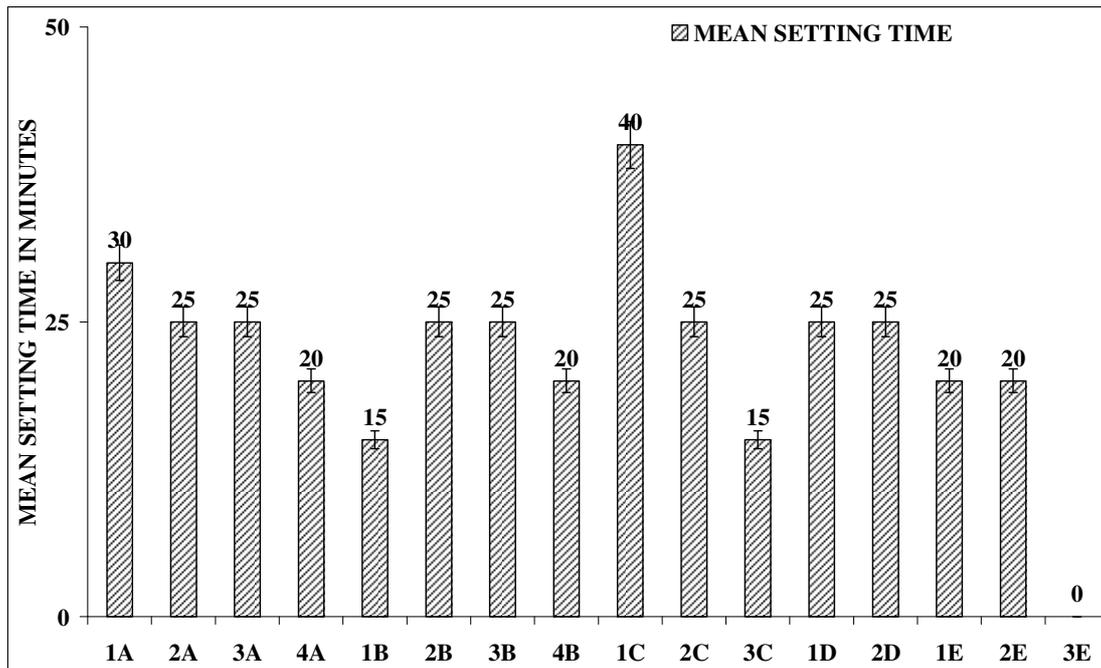


Figure 18. Mean setting time at core trials

The results of the one-off trial (core composition [1] trial cores subjected to twenty-four hours constant temperature 200 and 400 °C) to determine the time taken to remove moisture (visually inspected) from the core are shown in Figures 19 and 20. The weight loss temperatures were measured to an accuracy of 0.001%. The core weight loss was measured at their respective constant temperatures after every two hours. Two further batches fired at temperature 600 and 800 °C similarly gave a maximum weight loss within two hours. Since all other twenty-four hour test gave the same results, the test was not repeated for any further core trials.

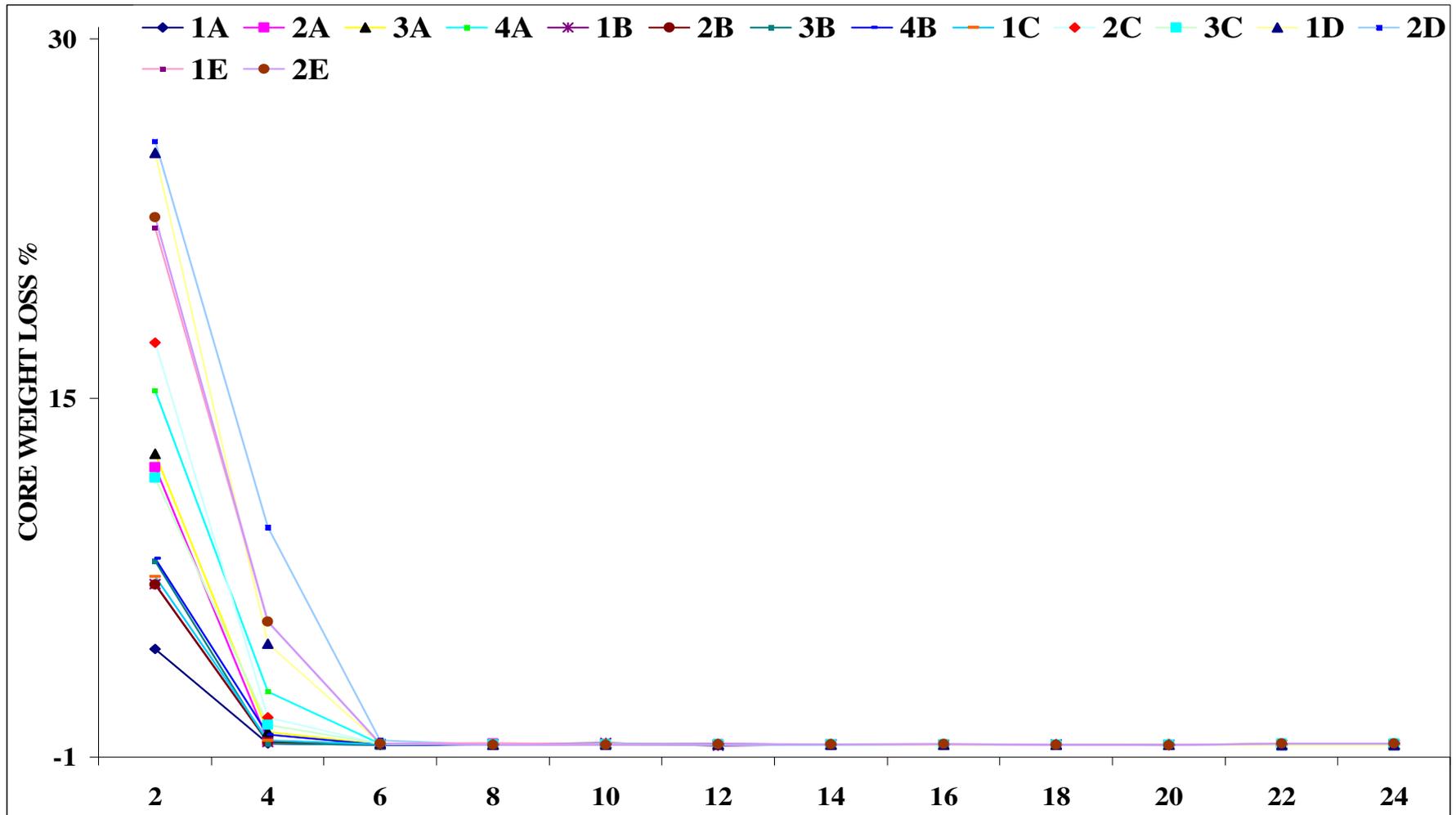


Figure 19. The 24 hours firing with weight loss on core trials at 200 °C

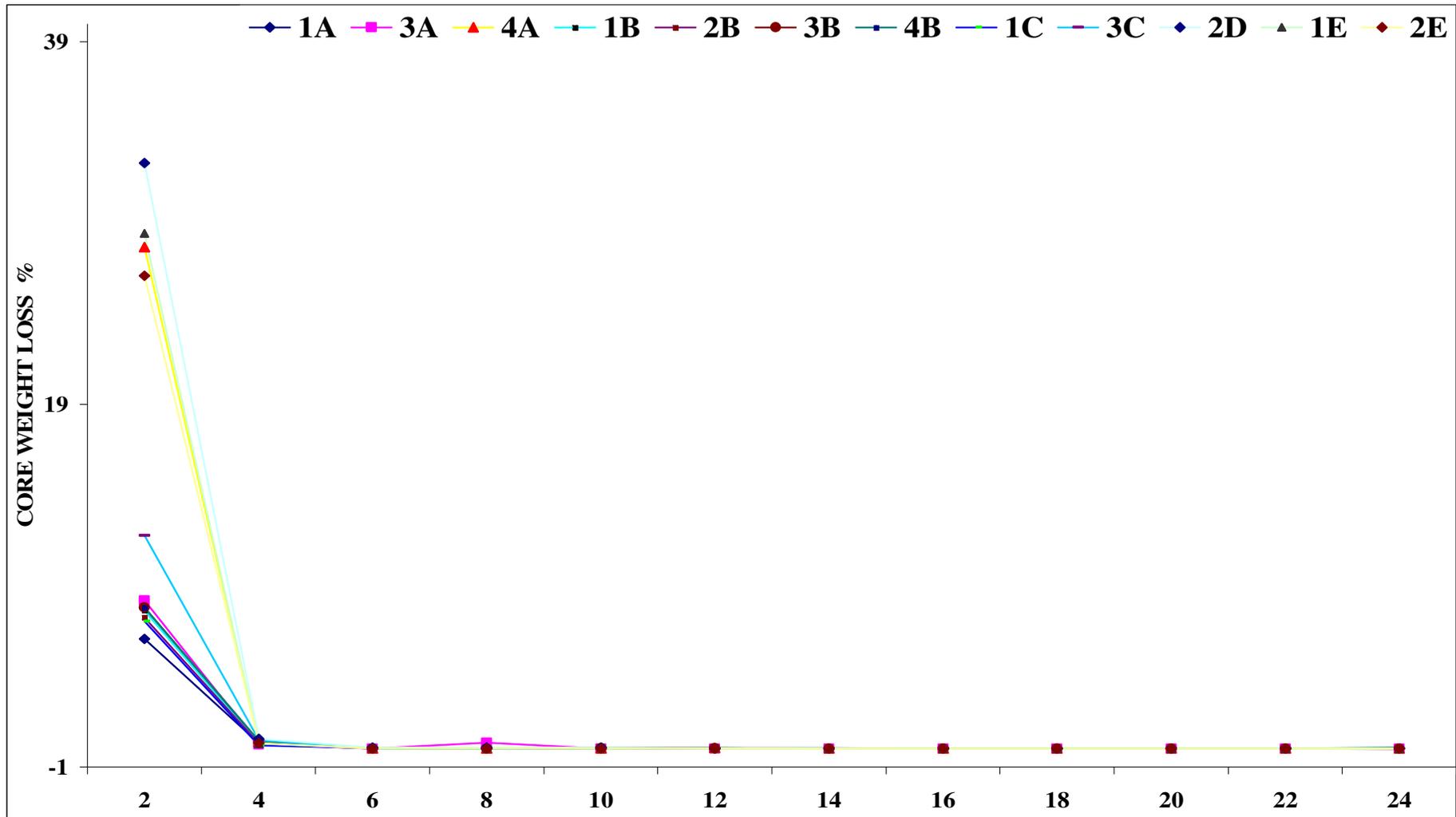


Figure 20. The 24 hours firing with weight loss on core trials at 400 °C

4.1.1 CORE COMPOSITION [1], MEAN MOR

The mean results of ten samples for each core trial tested using three-point bend testing are shown with error bars in Figures 21 to 25.

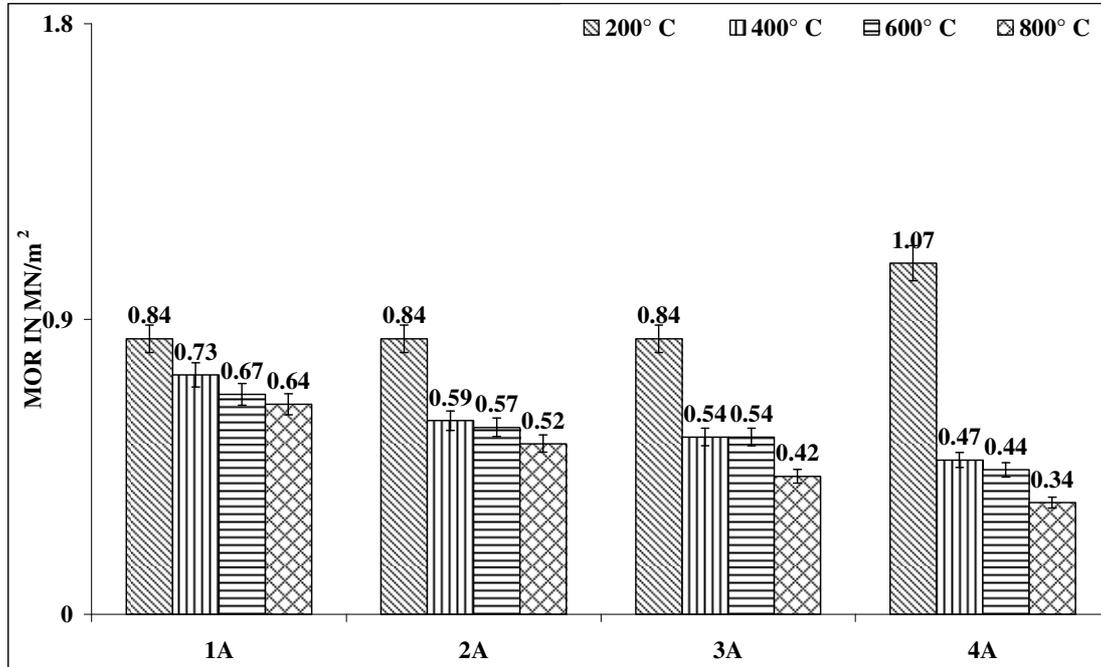


Figure 21. Mean MOR of Trial A at different fired temperatures

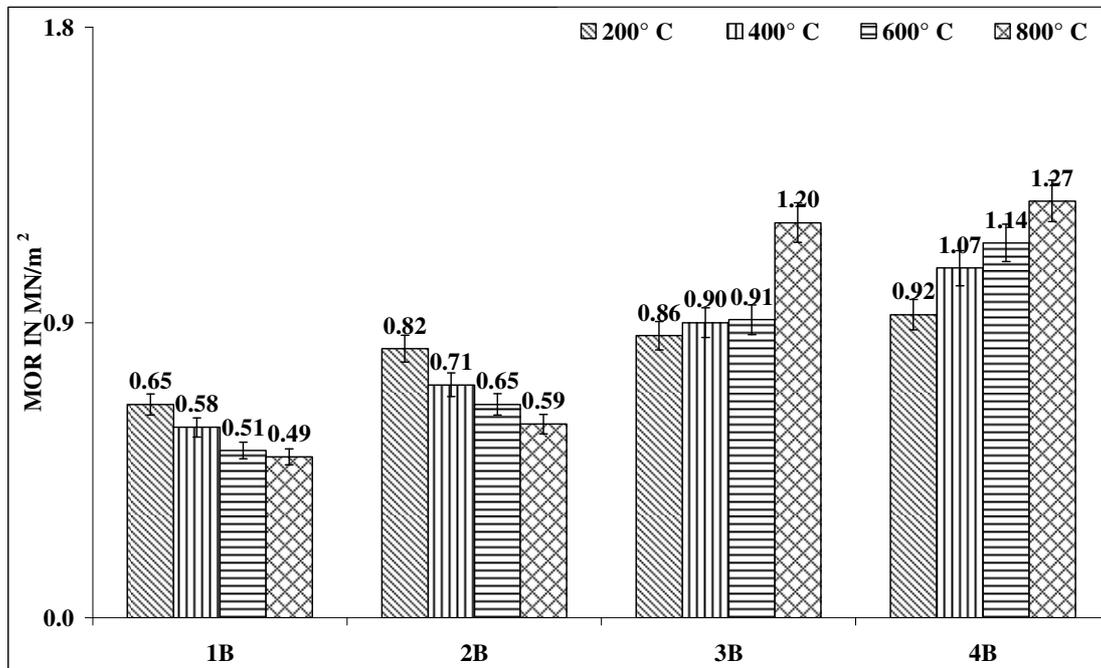


Figure 22. Mean MOR of Trial B at different fired temperatures

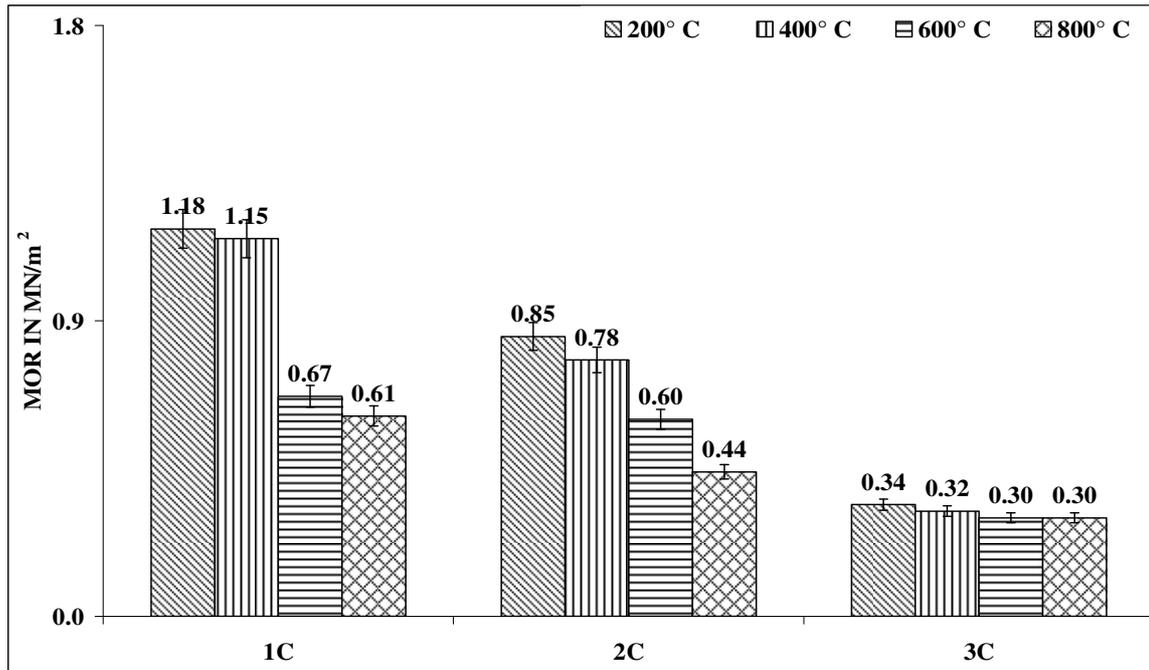


Figure 23. Mean MOR of Trial C at different fired temperatures

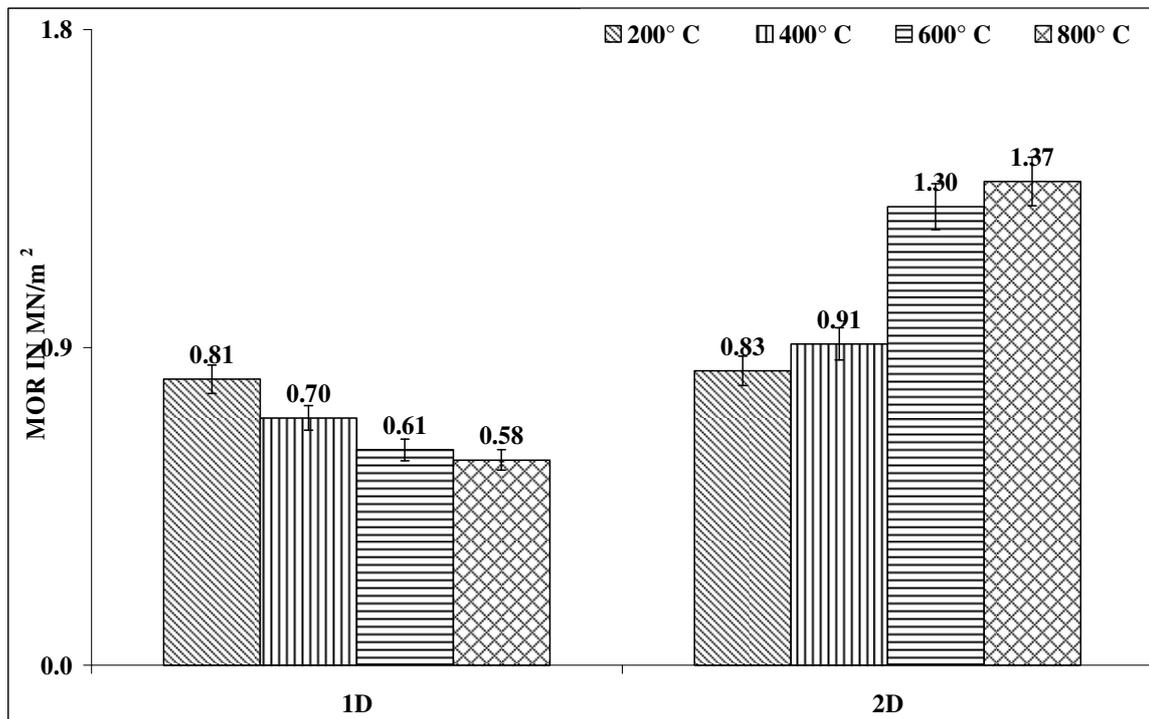


Figure 24. Mean MOR of Trial D at different fired temperatures

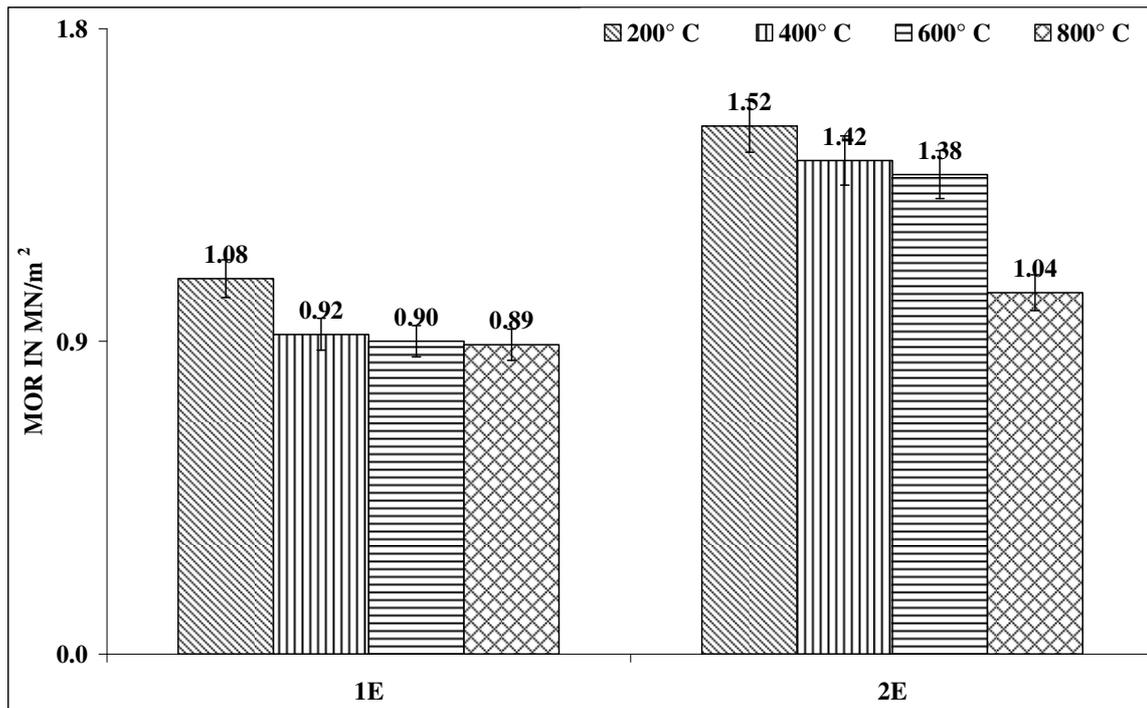


Figure 25. Mean MOR of Trial E at different fired temperatures

4.1.2 CORE REMOVAL

The assessment was visual, followed by probing with a blade. A single casting was done for each core (different core trials) at different fired temperatures. Figure 26 shows the effect of diluted nitric acid (1:10) within four hours on Trial 1E core fired at 400 °C. Results of core removal trials are shown in Table 27.



Figure 26. Effect of diluted nitric acid on Trial 1E core fired at 400 °C under four hours.

CORE REMOVAL FROM CASTING IN DILUTE NITRIC ACID(1:10)												
CORE FIRED TEMPERATURE	200 °C			400 °C			600 °C			800 °C		
TRIAL	1h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	O	x	✓	x	✓	✓	x	✓	✓	O	✓	✓
2A	O	O	✓	O	O	x	O	O	✓	O	x	✓
3A	O	O	x	O	x	✓	O	x	x	O	x	x
4A	O	x	✓	O	✓	✓	x	✓	✓	O	x	✓
1B	O	x	✓	x	✓	✓	x	✓	✓	O	x	✓
2B	O	x	✓	x	✓	✓	O	✓	✓	O	x	✓
3B	O	x	✓	x	✓	✓	O	✓	✓	O	x	✓
4B	O	x	✓	x	✓	✓	O	✓	✓	O	x	✓
1C	O	x	✓	O	O	✓	O	O	✓	O	x	x
2C	O	O	✓	O	O	✓	O	O	✓	O	x	✓
3C	O	O	✓	x	x	✓	O	x	✓	O	x	✓
1D	O	O	✓	O	x	✓	O	x	✓	O	x	✓
2D	O	x	✓	x	x	✓	O	x	✓	O	x	✓
1E	O	x	✓	x	✓	✓	x	✓	✓	O	✓	✓
2E	O	x	✓	x	✓	✓	x	✓	✓	O	✓	✓

O - No effect on core removal.

X- Some effect on core removal.

✓- Core completely collapses.

Table 27. Core removal in dilute nitric acid (1:10)

4.2 CORE COMPOSITION [2] RESULTS

The core trials were set with standard dry mix (without water/binder) for ten seconds and another twelve seconds slurry working life after mixing process (with binder/water). After mixing, air bubbles were removed before pouring into wooden core box. Figure 27 is shown in Section 5.1.2 (page 126). Figures 28, 29 and 30 are shown in Sections 5.1.3 and 5.1.4 (page 134). Figure 31 shows mean setting time in wooden core box for core trials.

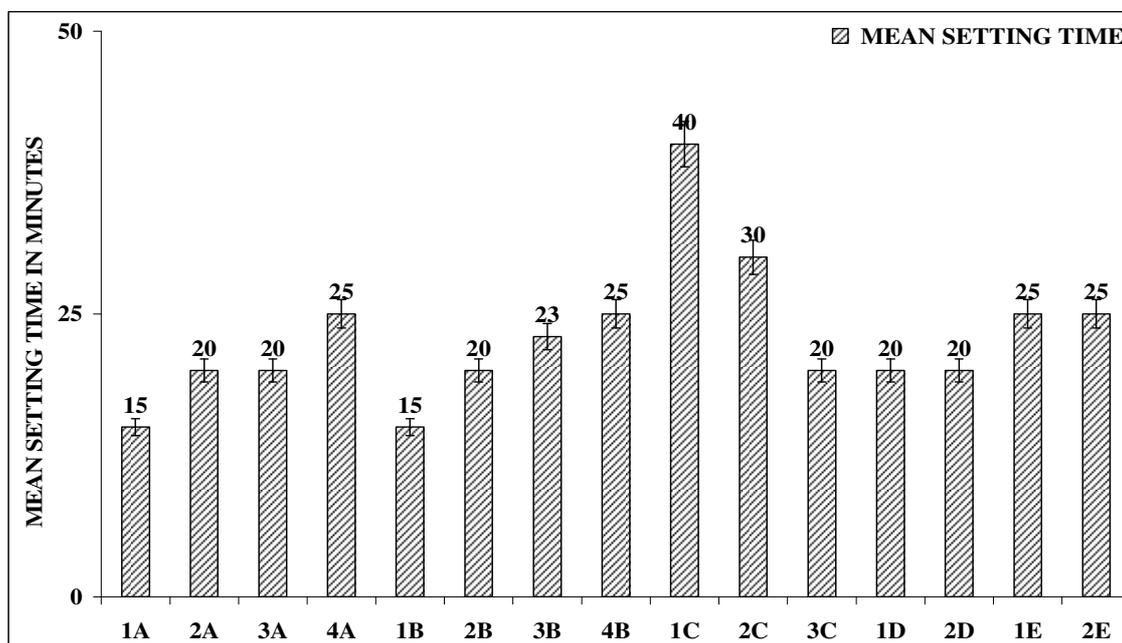


Figure 31. Mean setting time of core trials

On core composition [1], the core trials set very quickly since percentage of magnesium was one-percent. An experiment of adding less percentage of magnesium oxide to Trial 4A was carried out to find the effect on slurry working life. Figure 32 shows the slurry working life at different percentage of MgO added to Trial 4A. At 0.1% MgO, twelve seconds of working life were observed. The other core trials exhibited similar slurry working life.

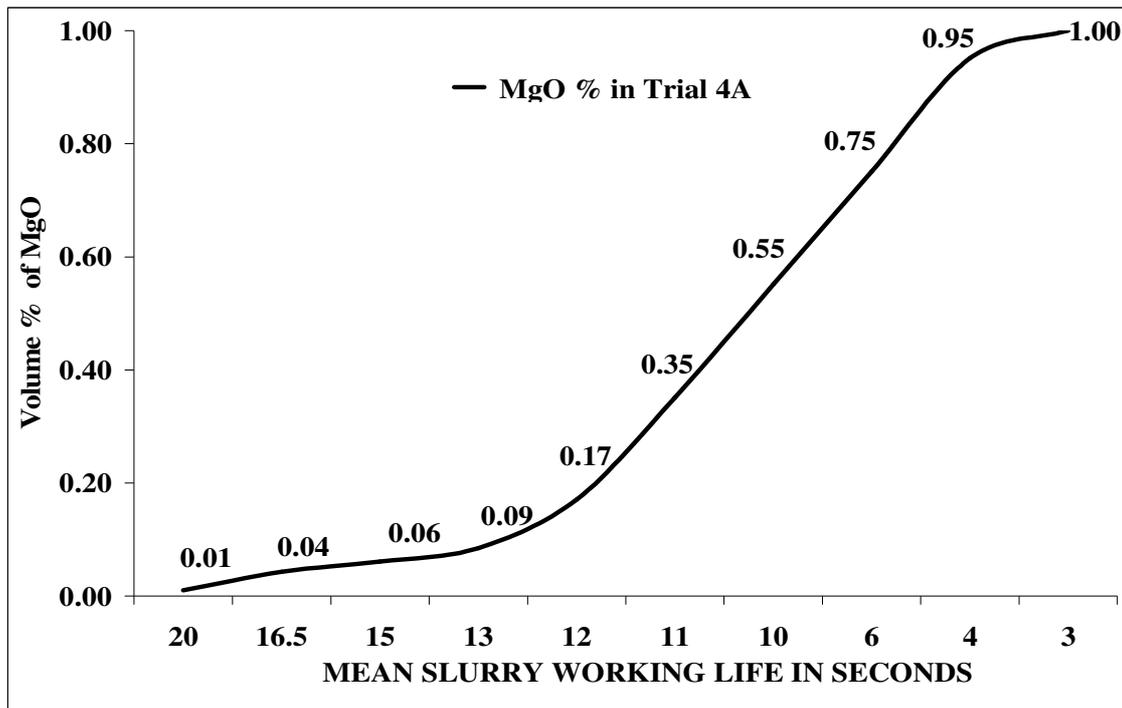


Figure 32. Mean slurry working life in Trial 4A at different percentages of MgO

4.2.1 CORE COMPOSITION [2], MEAN MOR

The mean results of ten samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 33 to 37.

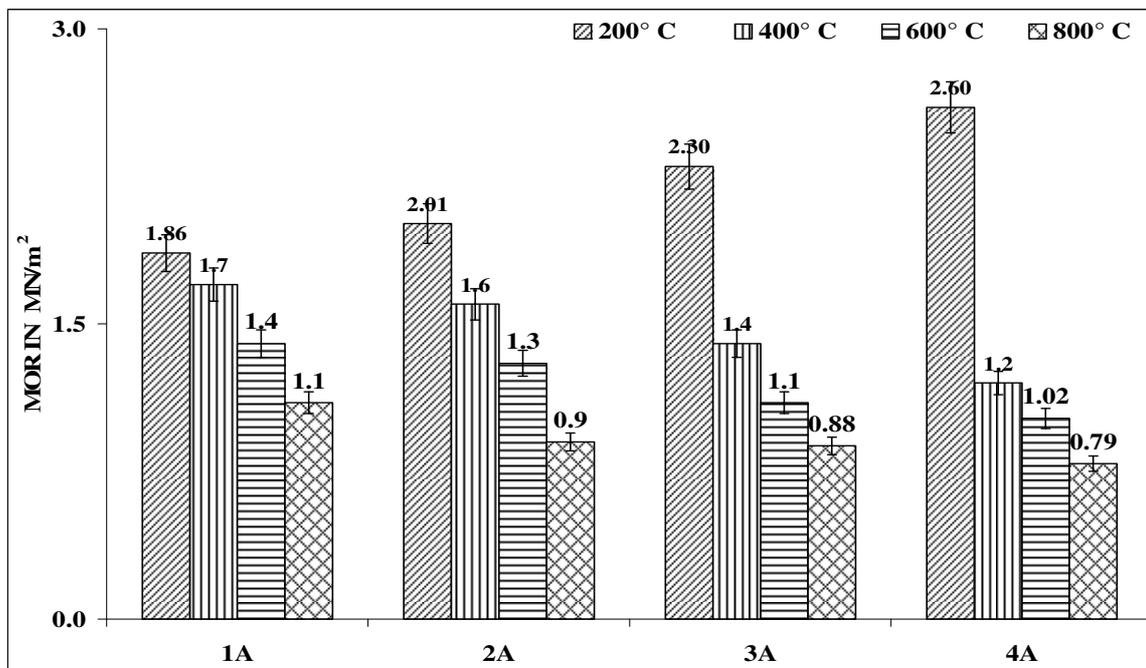


Figure 33. Mean MOR of Trial A at different fired temperatures

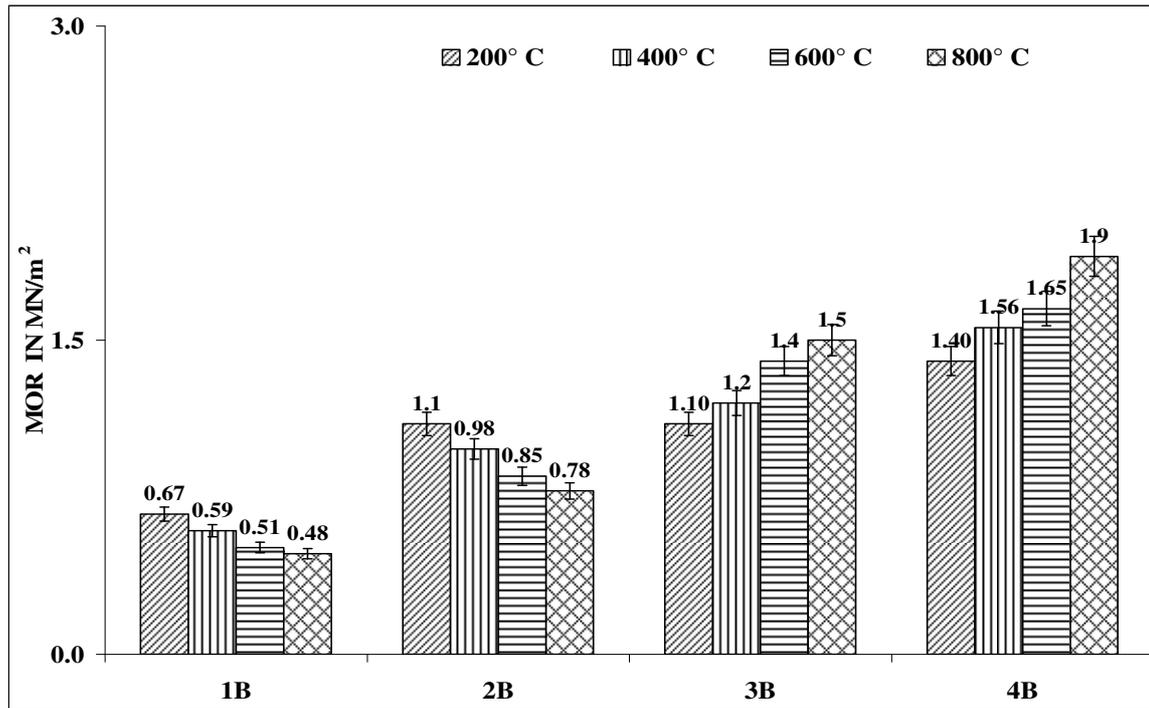


Figure 34. Mean MOR of Trial B at different fired temperatures

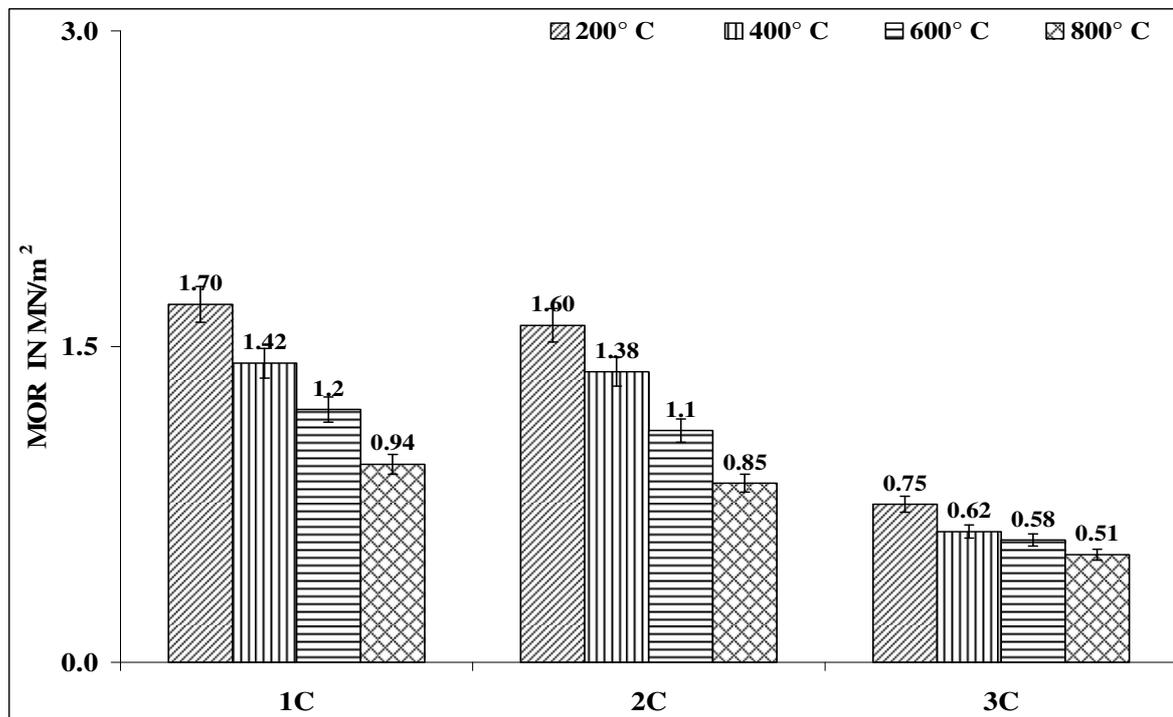


Figure 35. Mean MOR of Trial C at different fired temperatures

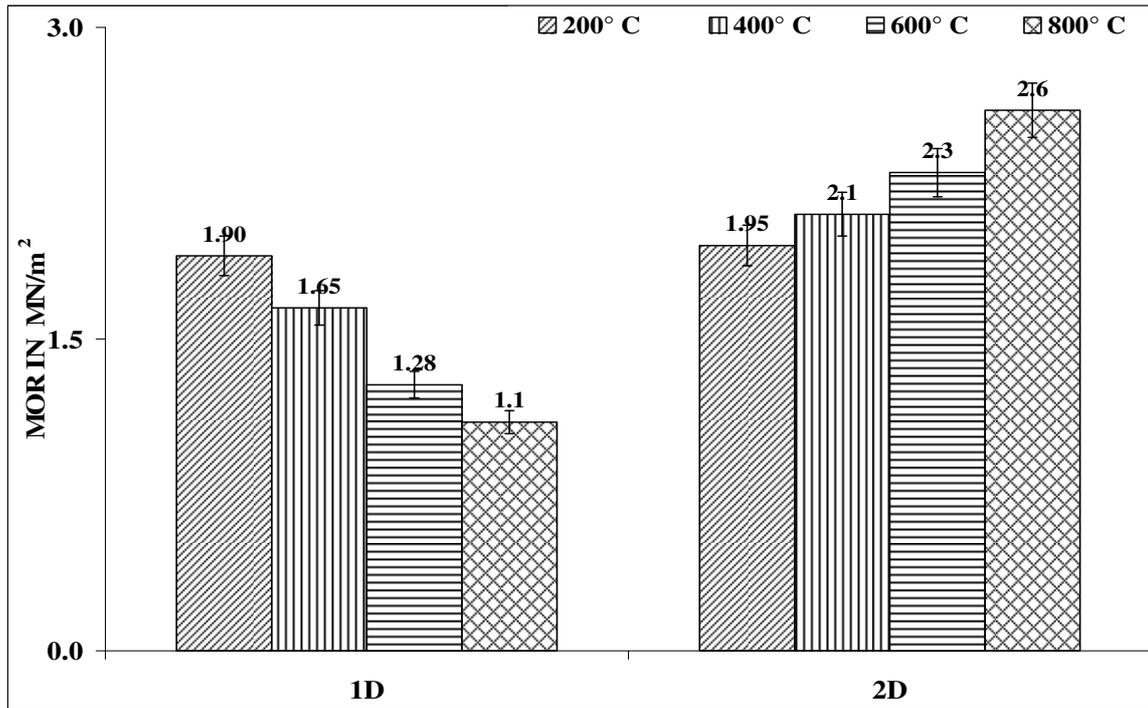


Figure 36. Mean MOR of Trial D at different fired temperatures

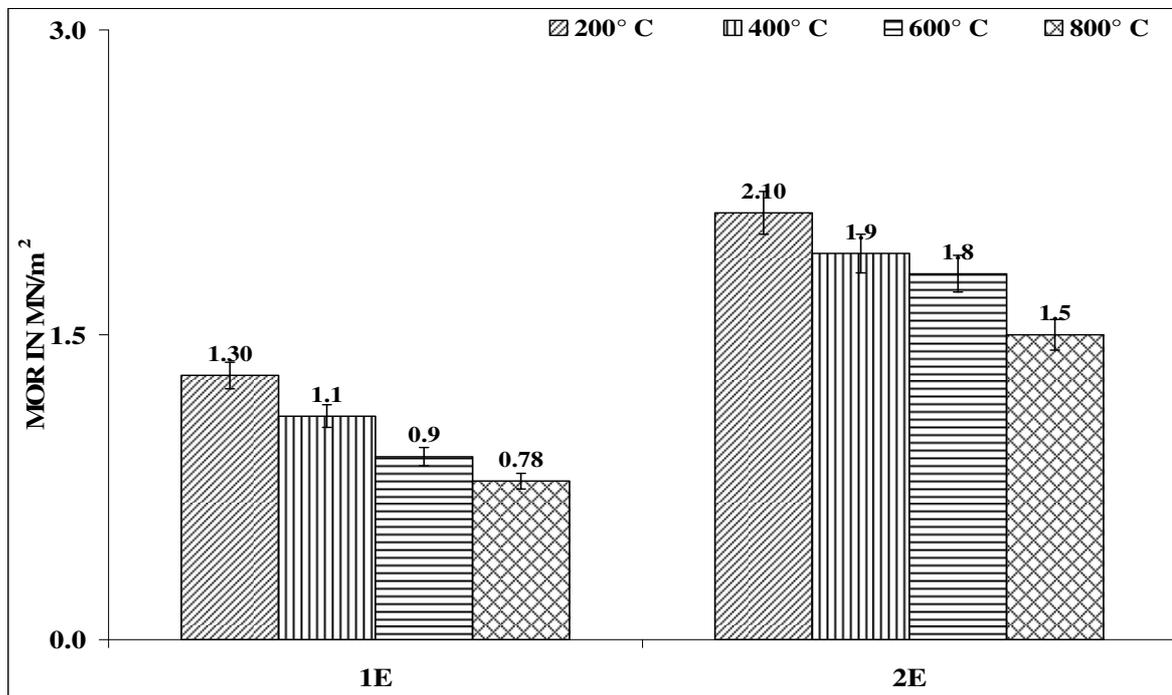


Figure 37. Mean MOR of Trial E at different fired temperatures

4.2.2 CORE REMOVAL

The assessment was visual followed by probing with a blade. A single castings were done for each core at different fired temperatures. Figures 38 to 39 shows the effect of dilute nitric acid (1:1, 1:2, 1:5) within four hours.



**Figure 38. Effect of dilute nitric acid (1:1, 1:2 and 1:5) on Trial 1B
fired at 200 °C after one hour**



**Figure 39. Effect of dilute nitric acid (1:1, 1:2 and 1:5) on Trial 1B
fired at 200 °C after four hours**

Higher concentration of nitric acid had a quick effect on cores which were fired at different temperatures and opposite effect for the less concentrated nitric acid. Results of other core removal are shown in Tables 28 to 30.

CORE REMOVAL FROM CASTING IN DILUTE NITRIC ACID (1:1,1:2 and 1:5)												
CORE FIRED TEMPERATURE	200 °C			400 °C			600 °C			800 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
2A	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
3A	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
4A	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
1B	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
2B	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
3B	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
4B	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
1C	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
2C	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
3C	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
1D	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
2D	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
1E	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓
2E	x	✓	✓	x	✓	✓	x	✓	✓	x	x	✓

X- Some effect on core removal.

✓- Core completely collapses.

Table 28. Effect of dilute nitric acid (1:1, 1:2, 1:5) on cores

CORE REMOVAL FROM CASTING IN DILUTE NITRIC ACID (1:50,1:100, 1:500)												
CORE FIRED TEMPERATURE	200 °C			400 °C			600 °C			800 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	O	x	✓	O	O	x	O	O	x	O	O	x
2A	O	O	✓	O	O	x	O	O	x	O	O	x
3A	O	O	x	O	x	x	O	O	x	O	O	x
4A	O	x	✓	O	O	x	O	O	x	O	O	x
1B	O	x	✓	O	x	✓	O	x	x	O	O	x
2B	O	x	✓	O	x	x	O	O	x	O	O	x
3B	O	O	x									
4B	O	O	x									
1C	O	O	✓	O	O	x	O	O	x	O	O	x
2C	O	O	✓	O	O	x	O	O	x	O	O	x
3C	O	O	✓	O	O	x	O	O	x	O	O	x
1D	O	O	x									
2D	O	O	x									
1E	O	O	x									
2E	O	x	x	O	O	x	O	O	x	O	O	x

O - No effect on core removal.

X- Some effect on core removal.

✓- Core completely collapses.

Table 29. Effect of dilute nitric acid (1:50, 1:100, 1:500) on cores

CORE REMOVAL FROM CASTING IN DILUTE NITRIC ACID (1:1000)												
CORE FIRED TEMPERATURE	200 °C			400 °C			600 °C			800 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	O	O	O	O	O	O	O	O	O	O	O	O
2A	O	O	O	O	O	O	O	O	O	O	O	O
3A	O	O	O	O	O	O	O	O	O	O	O	O
4A	O	O	O	O	O	O	O	O	O	O	O	O
1B	O	O	×	O	O	O	O	O	O	O	O	O
2B	O	O	O	O	O	O	O	O	O	O	O	O
3B	O	O	O	O	O	O	O	O	O	O	O	O
4B	O	O	O	O	O	O	O	O	O	O	O	O
1C	O	O	O	O	O	O	O	O	O	O	O	O
2C	O	O	O	O	O	O	O	O	O	O	O	O
3C	O	O	O	O	O	O	O	O	O	O	O	O
1D	O	O	O	O	O	O	O	O	O	O	O	O
2D	O	O	O	O	O	O	O	O	O	O	O	O
1E	O	O	O	O	O	O	O	O	O	O	O	O
2E	O	O	O	O	O	O	O	O	O	O	O	O

O - No effect on core removal.

X- Some effect on core removal.

Table 30. Effect of diluted nitric acid (1:1000) on core

4.3 CORE COMPOSITION [3] RESULTS

A twenty seconds dry mix (without water/binder) and another fifteen-second slurry working life after mixing process (with water/binders) was carried out. Air bubbles were removed before pouring into the wooden core box. Figure 42 Mean setting time in wooden core box. Figures 41 and 42 are shown in Sections 5.2.1 (page 136) and 5.2.3 (page 142).

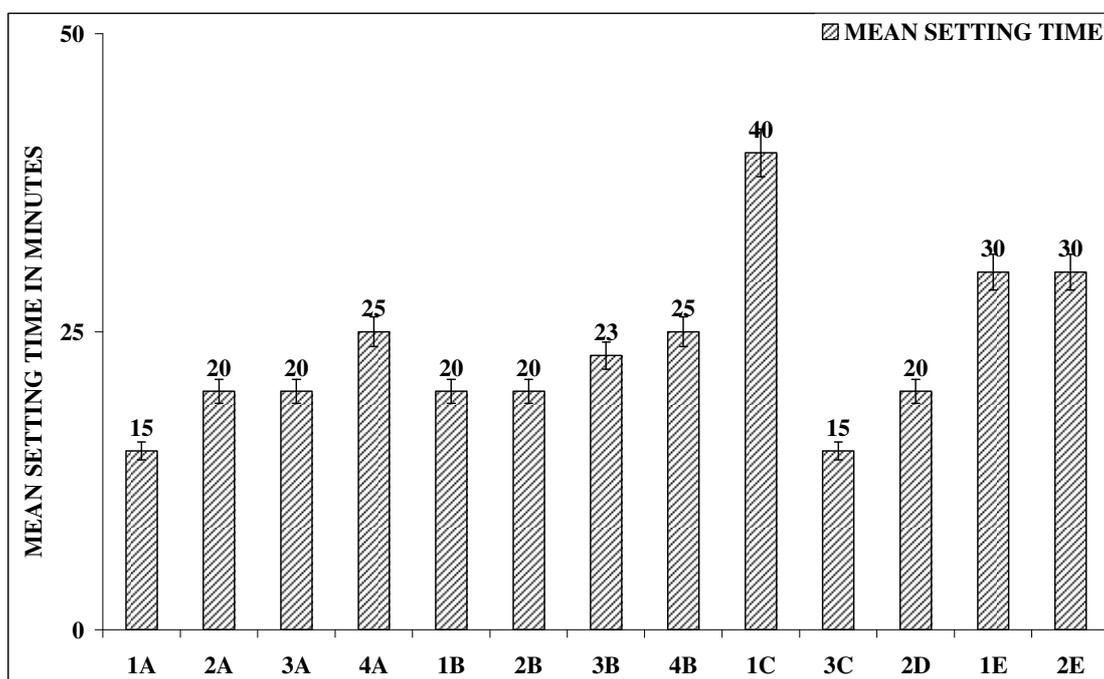


Figure 42. Mean setting time for core trials

4.3.1 CORE COMPOSITION [3], MEAN MOR

The mean results of 25 samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 43 to 47.

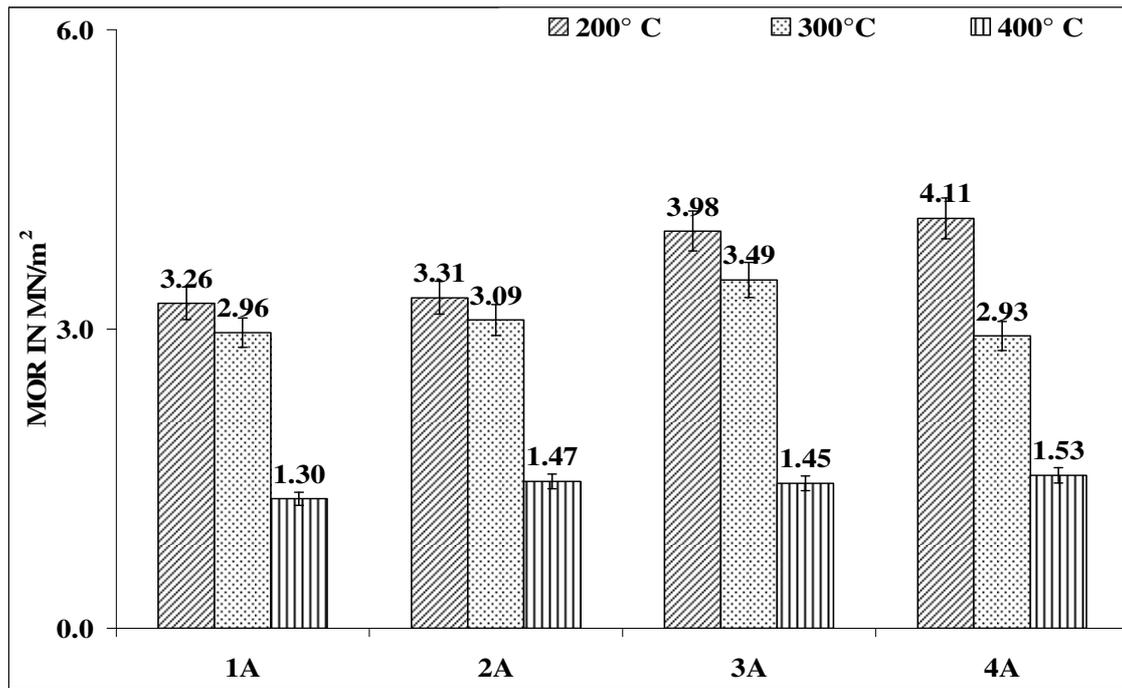


Figure. 43 Mean MOR of Trial A at different fired temperatures

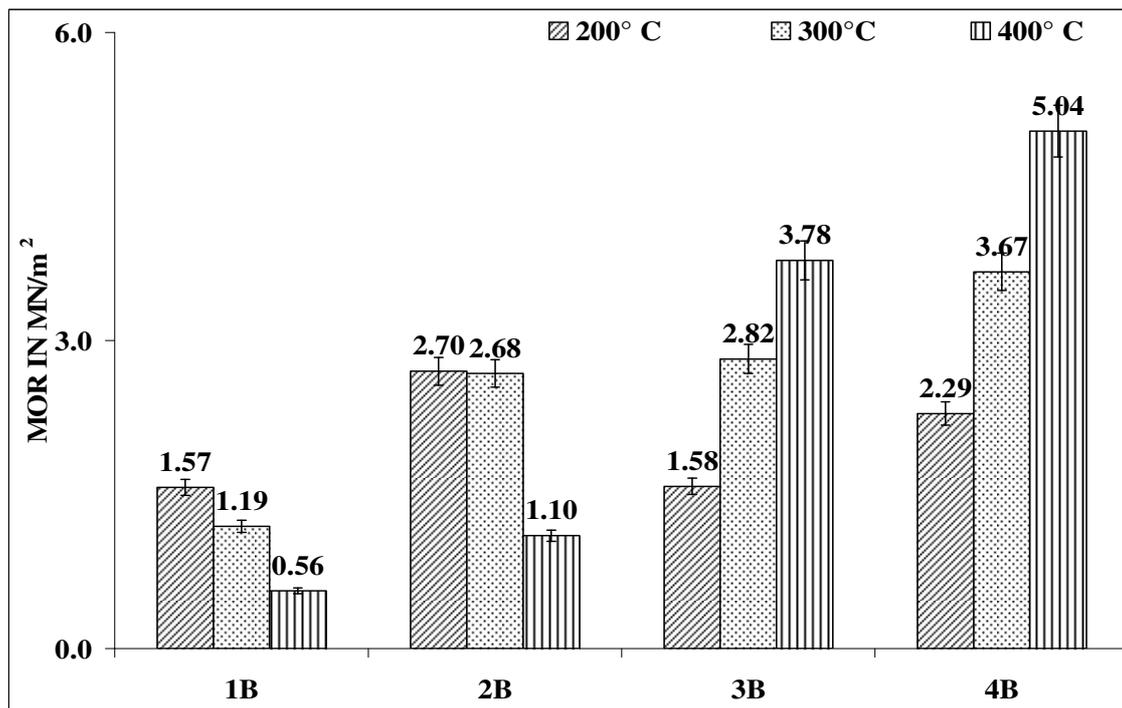


Figure. 44 Mean MOR of Trial B at different fired temperatures

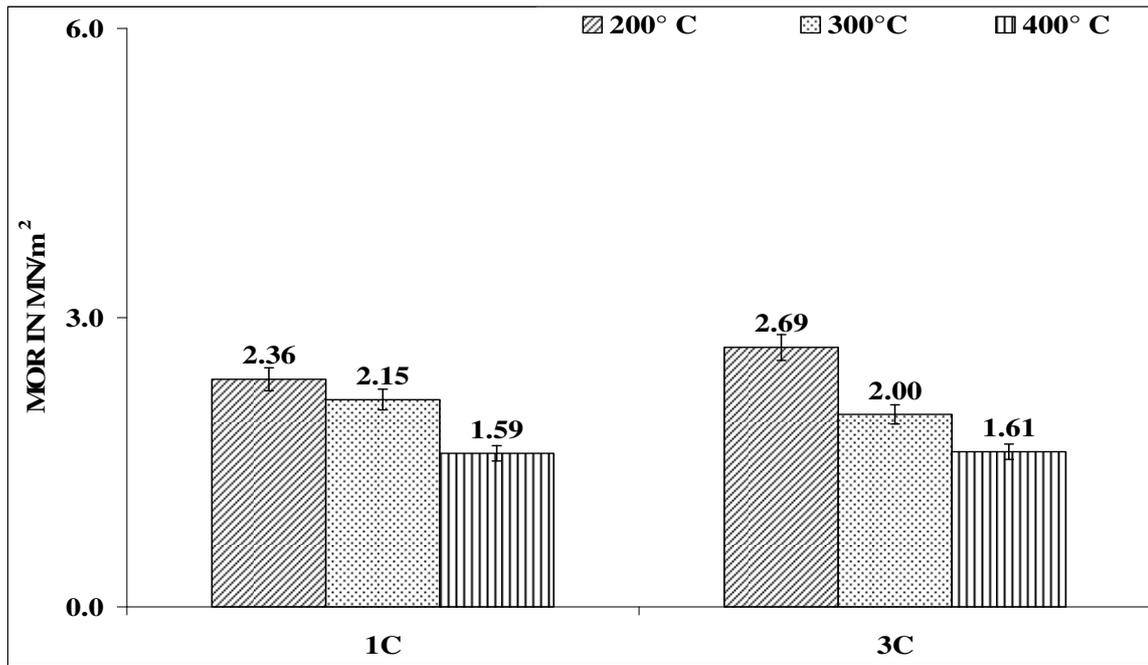


Figure. 45 Mean MOR of Trial C at different fired temperatures

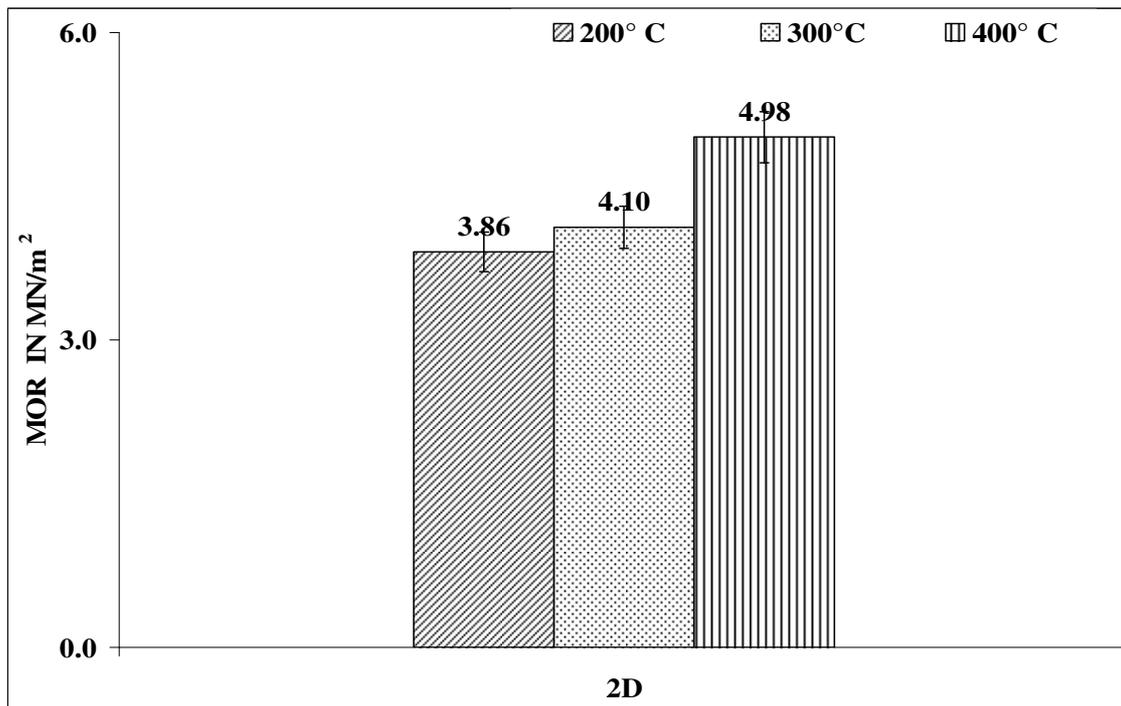


Figure. 46 Mean MOR of Trial D at different fired temperatures

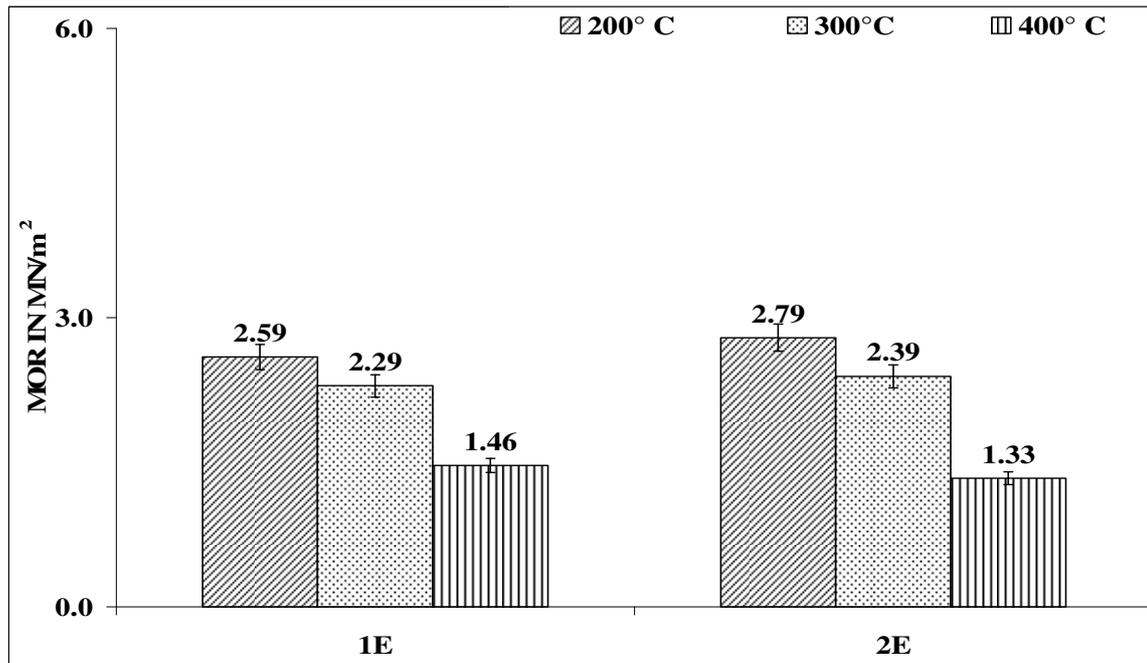


Figure. 47 Mean MOR of Trial E at different fired temperatures

4.3.2 SEM PICTURE ON CORE TRIAL

Trial 4A core surfaces fired at 200 °C were examined using scanning electron microscopy (SEM). The images of Trial 4A surface with different fused silica mesh size used in core composition [3] and [1] are shown in Figures 48 and 49

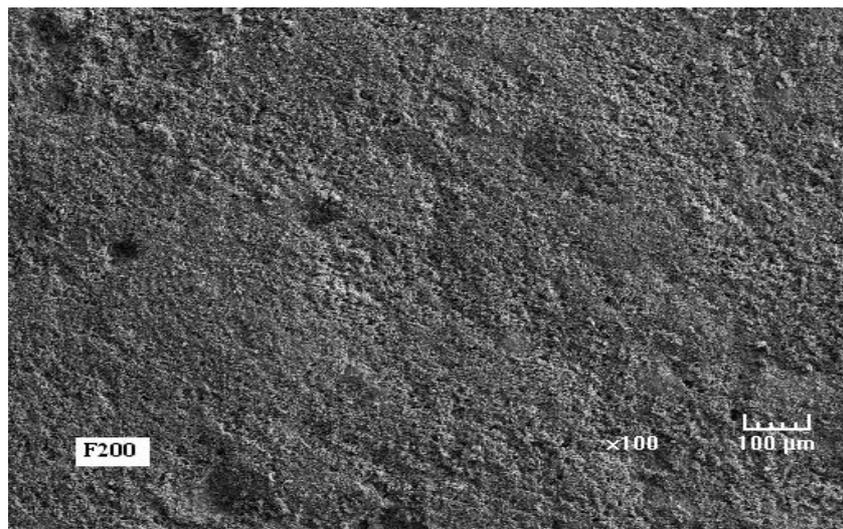


Figure 48. Trial 4A core surface with Fused silica mesh size (-200/-120) fired at 200 °C.

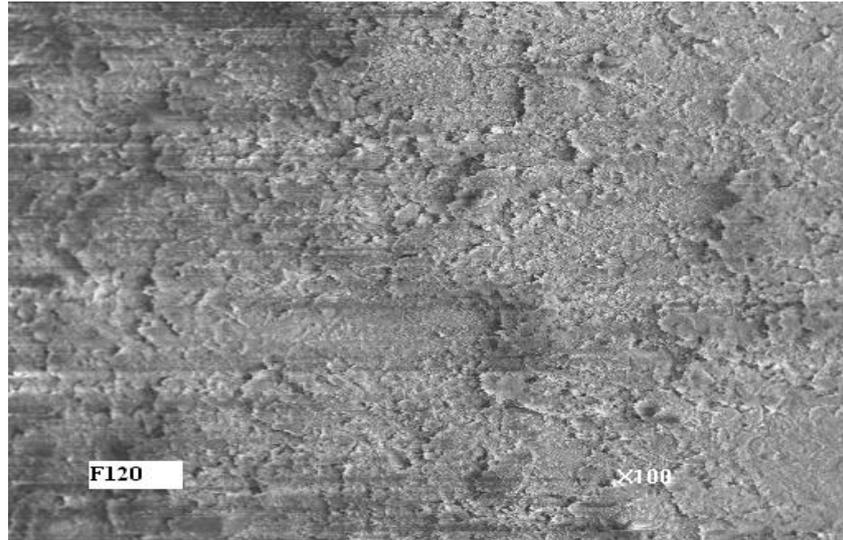


Figure 49. Trial 4A core surface with Fused silica mesh size (-120/30-50) fired at 200 °C.

4.3.3 CORE REMOVAL

The assessment was visual followed by probing with a blade. A single casting was carried out for each core fired at different temperatures. Figure 50 shows the effect of dilute potassium hydroxide (1:100) on Al-LM25.



Figure 50. Al-LM25 attacked by dilute potassium hydroxide.



Figure 51. Trial 1B core leached by dilute citric acid (1:1) at 200 °C.

Diluted acetic and citric acids were used to remove the cores. Tables 31 to 33 show other effects of dilute acids for cores fired at different temperatures.

CORE REMOVAL FROM CASTING IN DILUTE ACETIC AND CITRIC ACID (1:1)									
CORE FIRED TEMPERATURE	200 °C			300 °C			400 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	x	✓	✓	x	x	✓
2A	x	✓	✓	x	✓	✓	x	x	✓
3A	x	✓	✓	x	✓	✓	x	x	✓
4A	x	✓	✓	x	✓	✓	x	x	✓
1B	x	✓	✓	x	✓	✓	x	x	✓
2B	x	✓	✓	x	✓	✓	x	x	✓
3B	x	✓	✓	x	✓	✓	x	x	✓
4B	x	✓	✓	x	✓	✓	x	x	✓
1C	x	✓	✓	x	✓	✓	x	x	✓
3C	x	✓	✓	x	✓	✓	x	x	✓
2D	x	✓	✓	x	✓	✓	x	x	✓
1E	x	✓	✓	x	✓	✓	x	x	✓
2E	x	✓	✓	x	✓	✓	x	x	✓

O - No effect on core removal

x- Some effect on core removal

✓- Core completely collapses.

Table31. Effect of dilute acetic and citric acid (1:1) on cores

CORE REMOVAL FROM CASTING IN DILUTE ACETIC ACID (1:100)									
CORE FIRED TEMPERATURES	200 °C			300 °C			400 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	x	✓	✓	O	x	x
2A	x	✓	✓	x	✓	✓	x	✓	✓
3A	x	✓	✓	x	x	✓	x	✓	✓
4A	O	x	✓	x	x	✓	x	✓	✓
1B	✓	✓	✓	✓	✓	✓	✓	✓	✓
2B	✓	✓	✓	✓	✓	✓	✓	✓	✓
3B	x	✓	✓	x	x	✓	x	x	✓
4B	x	✓	✓	O	x	✓	O	x	✓
1C	x	x	✓	x	✓	✓	x	x	✓
3C	x	x	✓	O	x	✓	O	x	✓
2D	x	✓	✓	x	x	✓	x	x	✓
1E	O	x	✓	O	x	x	O	x	x
2E	O	x	✓	O	x	x	O	x	x

O - No effect on core removal

x- Some effect on core removal

✓- Core completely collapses.

Table32. Effect of dilute acetic acid (1:100) on cores

CORE REMOVAL FROM CASTING IN DILUTE CITRIC ACID (1:100)									
CORE FIRED TEMPERATURES	200 °C			300 °C			400 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	x	x	x	O	O	x
2A	x	x	✓	O	x	x	O	O	x
3A	x	x	✓	O	x	x	O	O	x
4A	O	x	✓	O	x	x	O	O	x
1B	✓	✓	✓	✓	✓	✓	✓	✓	x
2B	x	✓	✓	x	✓	✓	x	✓	x
3B	x	x	✓	O	x	x	O	x	x
4B	O	x	✓	O	x	x	O	x	x
1C	O	x	✓	O	x	x	O	O	x
3C	O	x	✓	O	x	x	O	O	x
2D	O	x	✓	O	x	x	O	O	x
1E	x	x	✓	O	x	x	O	O	x
2E	O	x	✓	O	x	x	O	x	x

O - No effect on core removal

x- Some effect on core removal

✓- Core completely collapses.

Table 33. Effect of dilute citric acid (1:100) on cores

4.4 CORE COMPOSITION [4] RESULTS

The standard thirty-second dry mix (without water/binder) and another thirty-five second of mean slurry working life after mixing (with water/binder) were adopted for all core trials. Figure 52 Mean setting time of core trials.

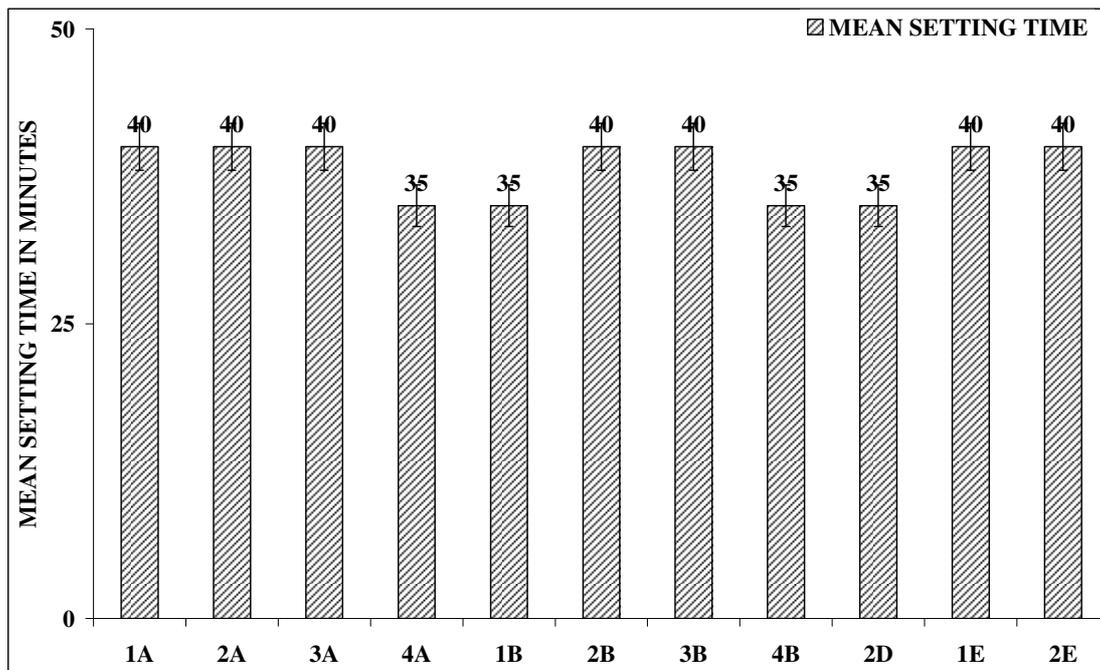


Figure 52. Mean setting time of core trials

A batch of 25 cores was made with core trials. It was left to dry out for 24 hours at room temperature in an open box to remove any excess moisture content in cores. The cores were fired to 200 °, 400 °, 600 ° and 800 °C. After the firing process, they were left to cool to room temperature for two hours.

4.4.1 CORE COMPOSITION [4] MEAN DIMENSION CHANGES

The core trial batch dimensions (diameter and length) were recorded before and after firing the cores. The dimensions were recorded after cooling for two hours to room temperature from respective firing temperature. Figures 53 to 56 show mean percentage length contraction at different fired temperatures.

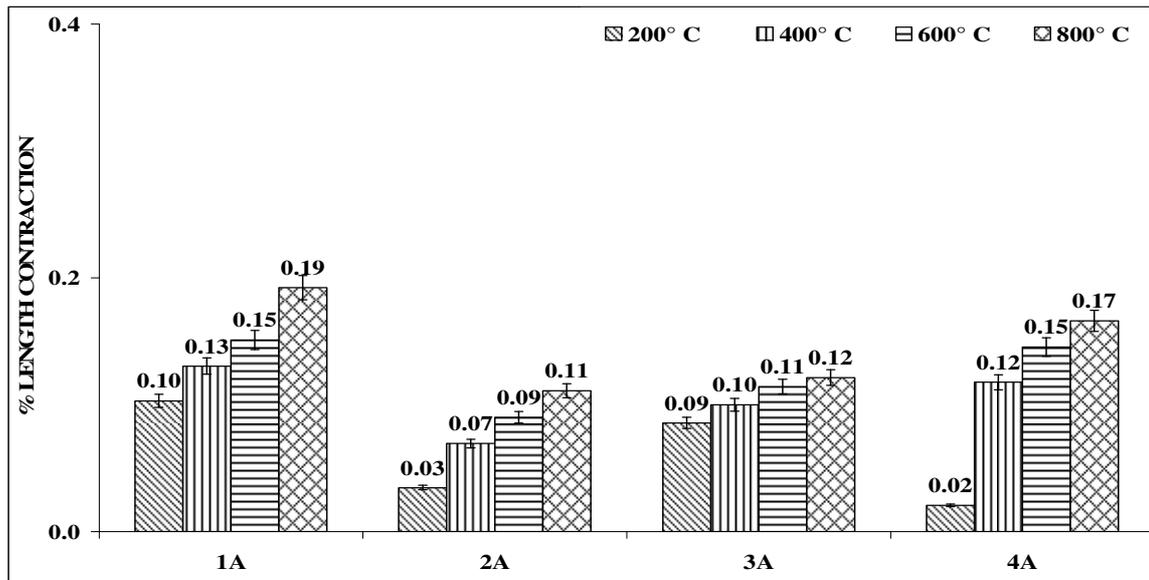


Figure 53. Mean percentage length contraction of Trial A at different fired temperature

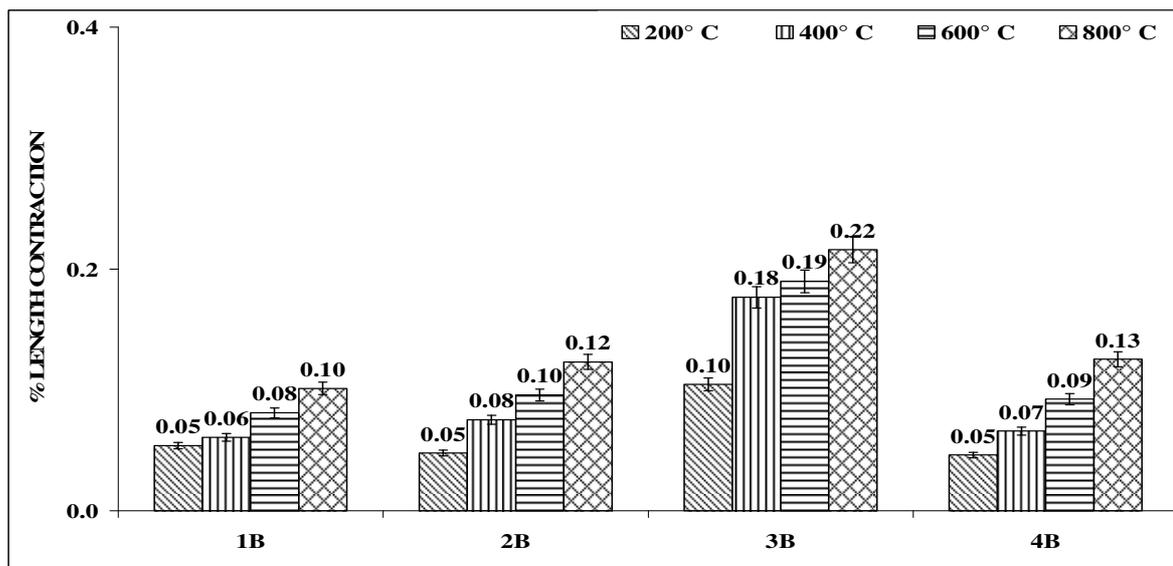


Figure 54. Mean percentage length contraction of Trial B at different fired temperatures

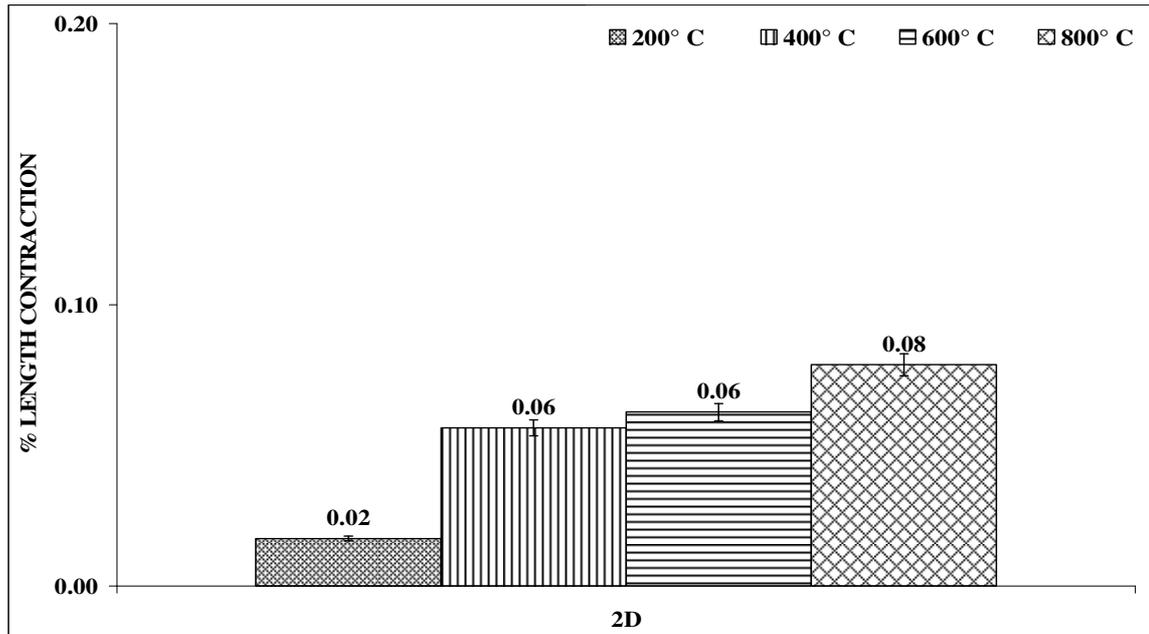


Figure 55. Mean percentage length contraction of Trial D at different fired temperatures

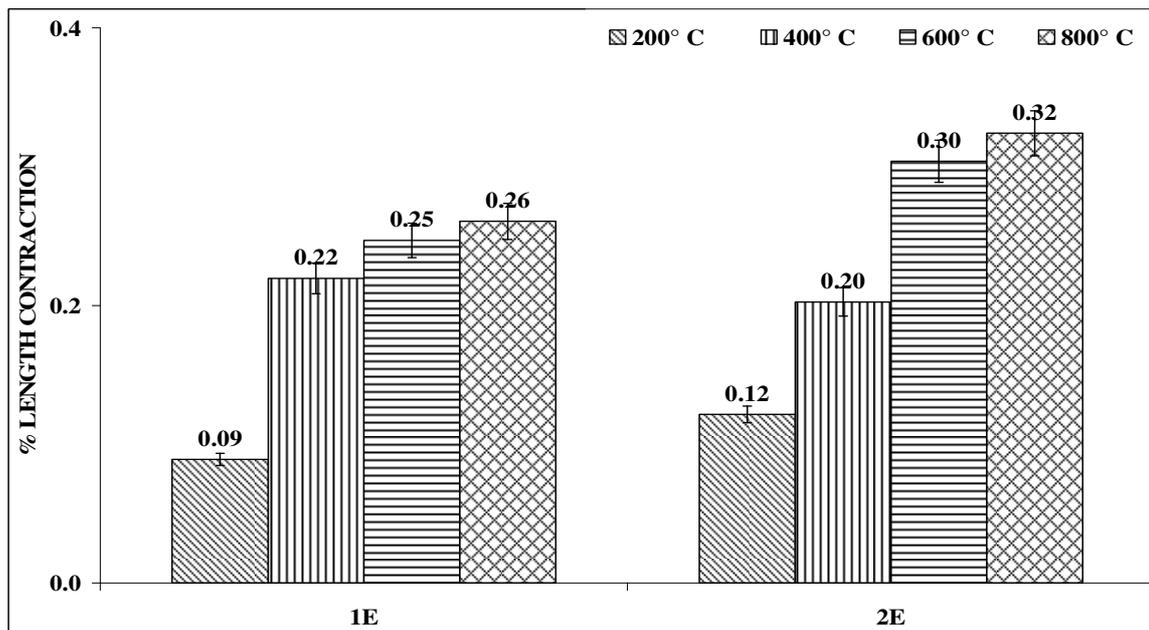


Figure 56. Mean percentage length contraction of Trial E at different fired temperatures

Figures 57 to 60 show the mean percentage of diameter shrinkage at different temperatures

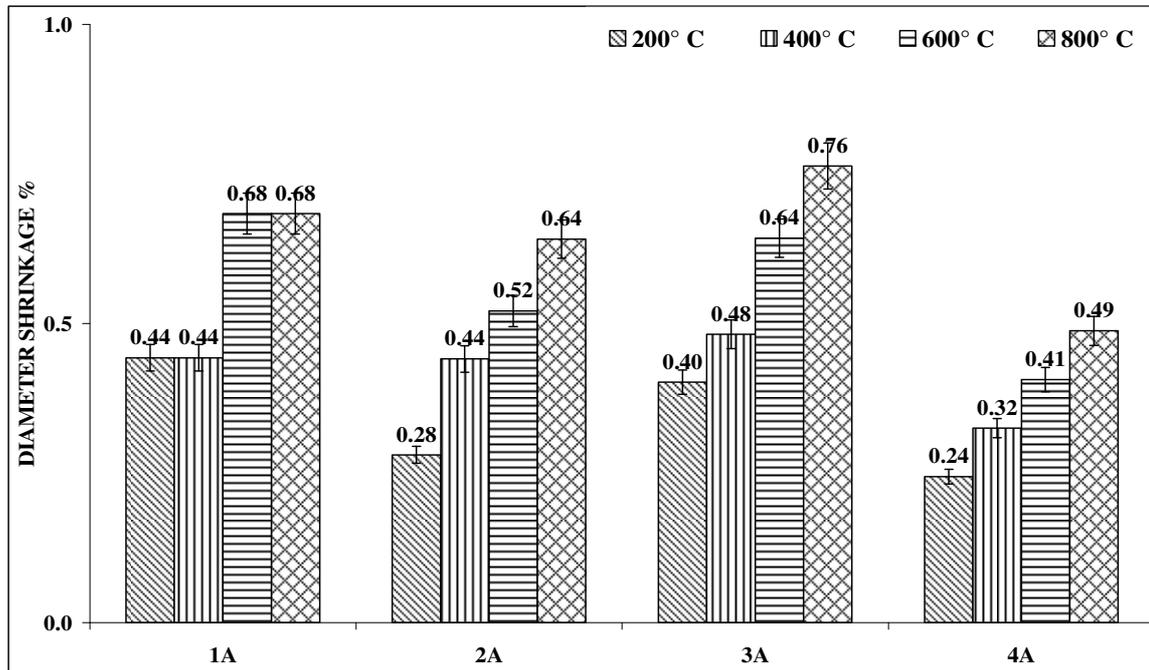


Figure 57. Mean diameter shrinkage percentage of Trial A at different fired temperatures

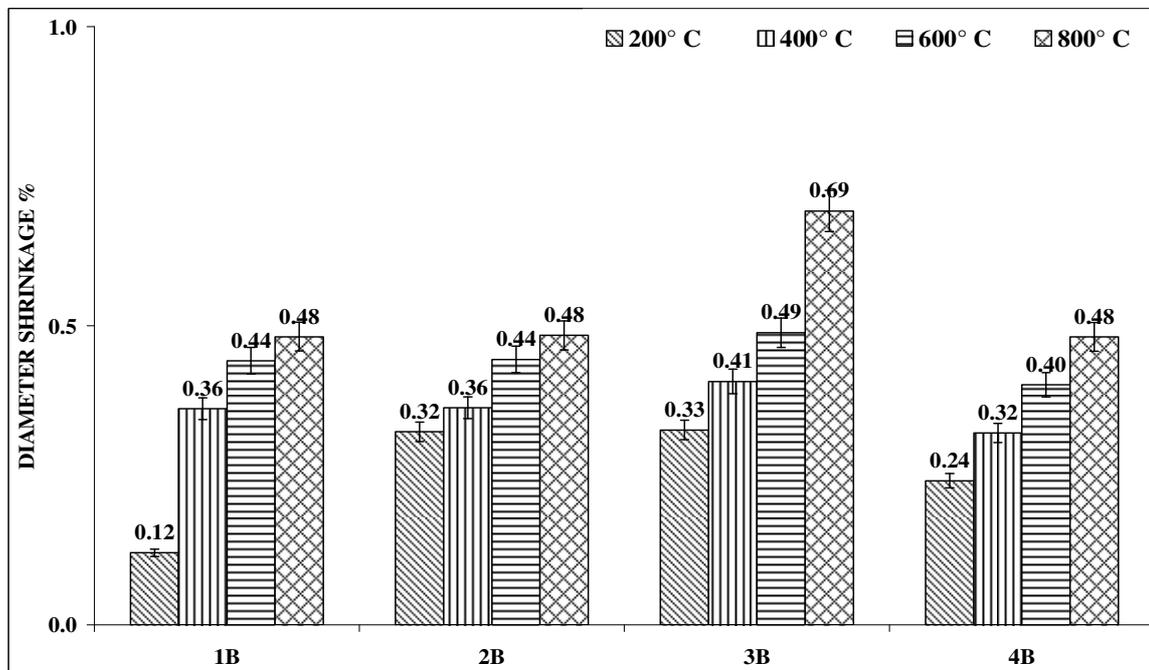


Figure 58. Mean diameter shrinkage percentage of Trial B at different fired temperatures

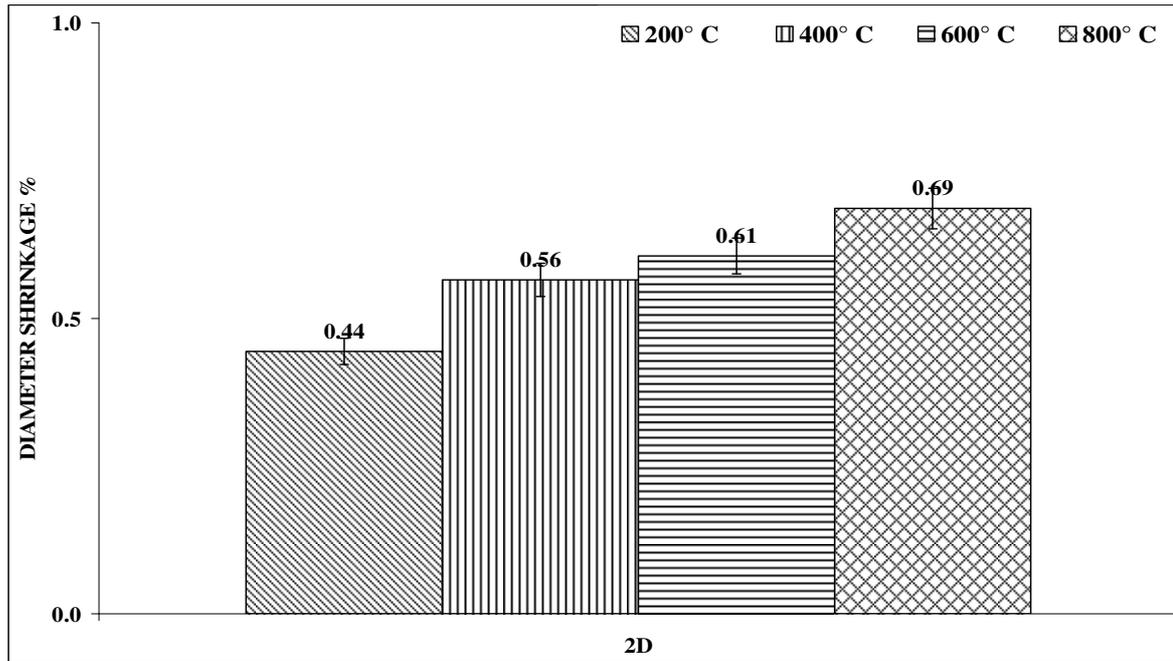


Figure 59. Mean diameter shrinkage percentage of Trial D at different fired temperatures

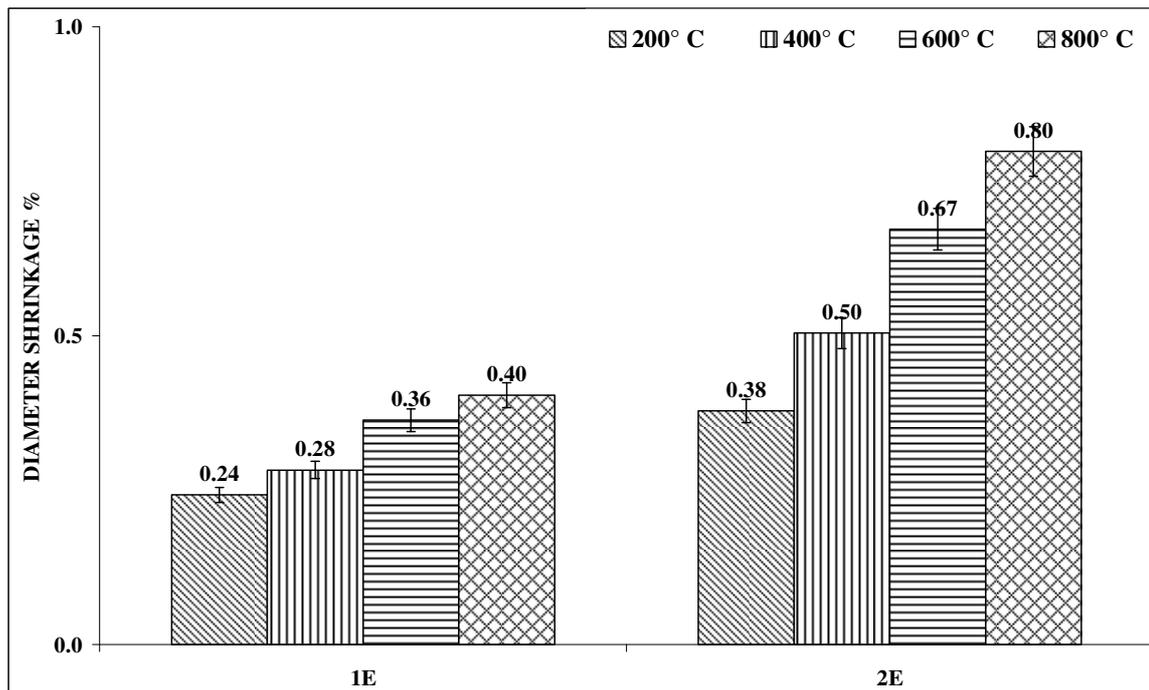


Figure 60. Mean diameter shrinkage percentage of Trial E at different fired temperatures

4.4.2 CORE COMPOSITION [4], MEAN MOR

The mean results of 25 samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 61 to 64.

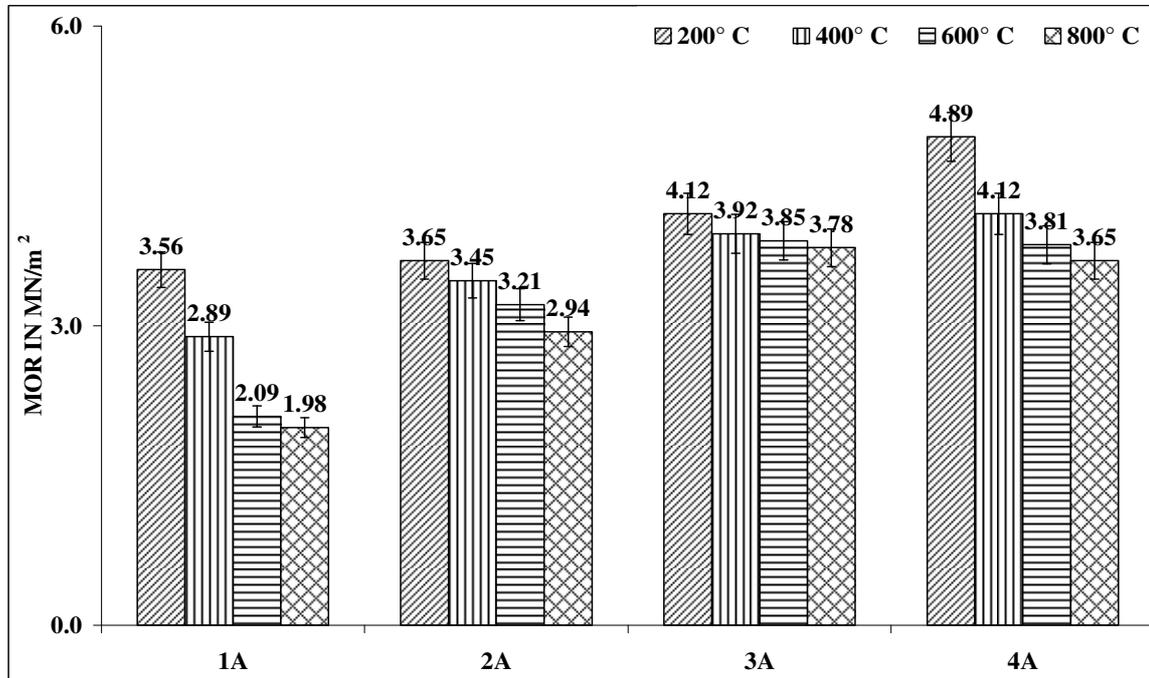


Figure 61. Mean MOR of Trial A at different fired temperatures

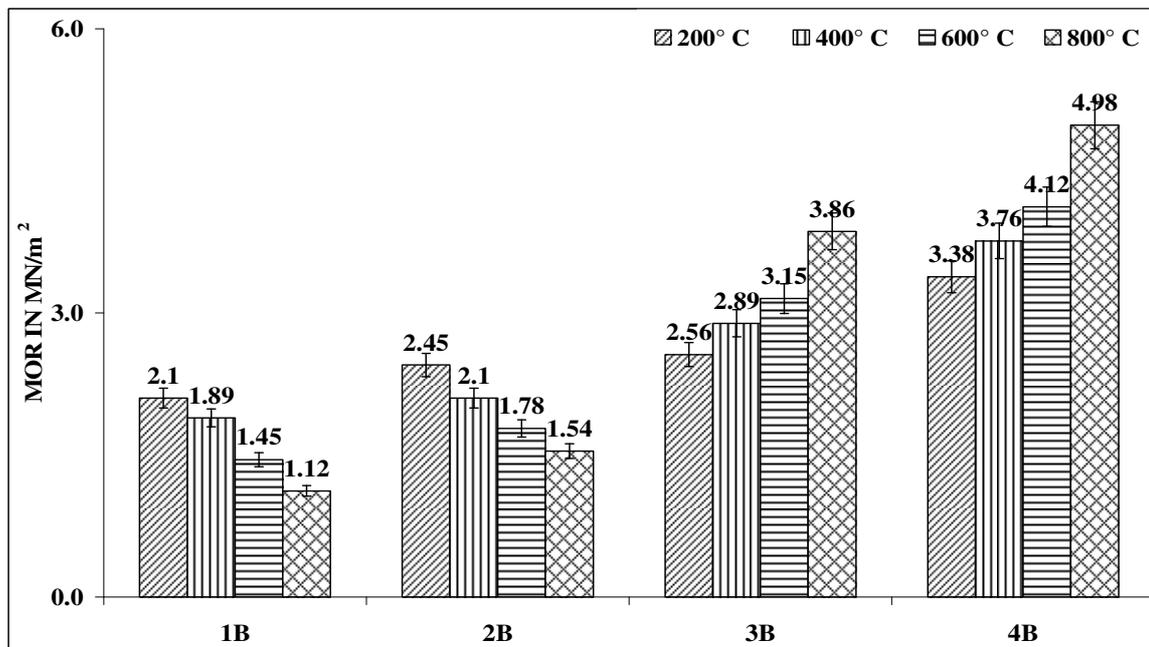


Figure 62. Mean MOR of Trial B at different fired temperatures

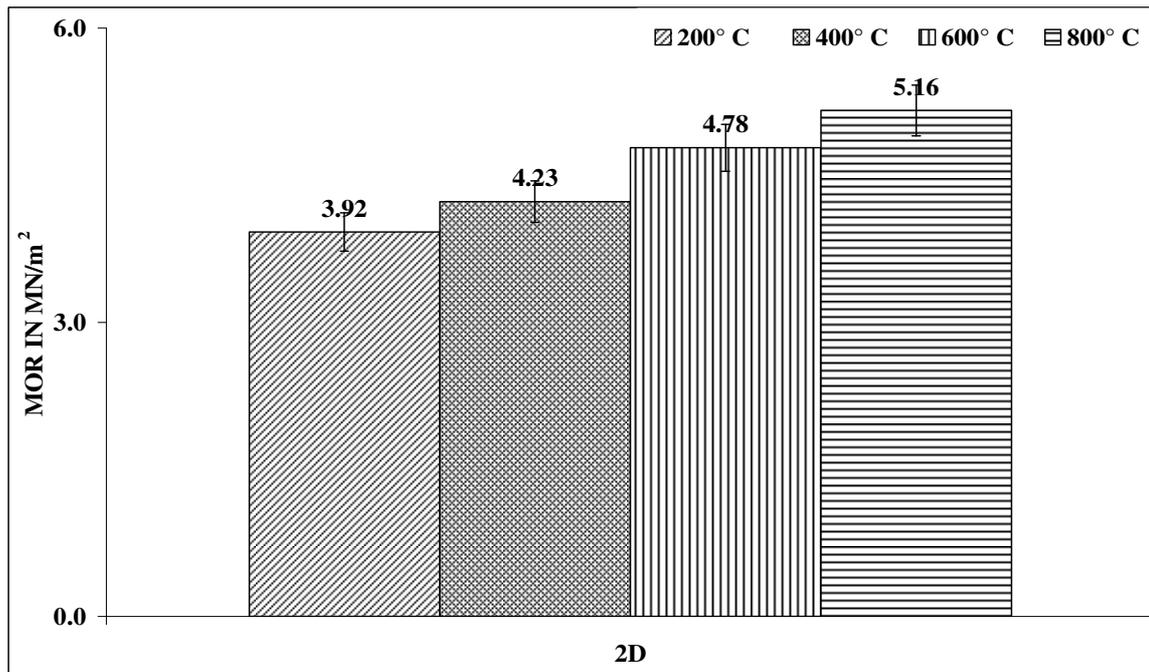


Figure 63. Mean MOR of Trial D at different fired temperatures

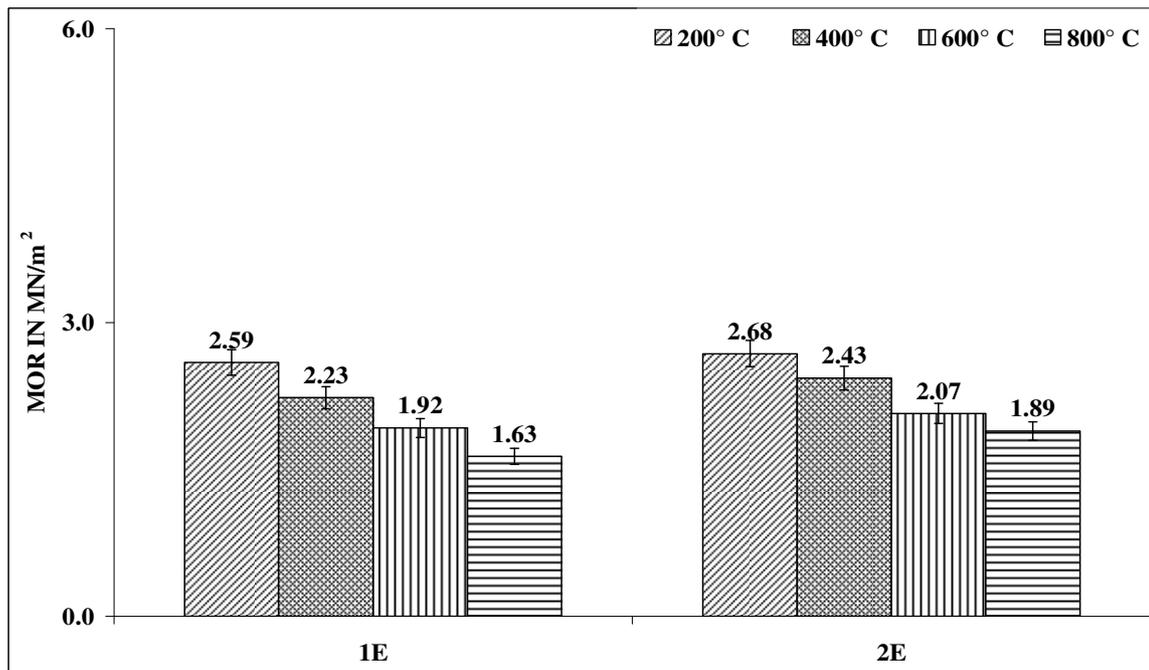


Figure 64. Mean MOR of Trial E at different fired temperatures

4.4.3 CORE REMOVAL

The assessment of removal was visual followed by probing with a blade. A single casting was carried out for each core fired at different temperatures; Table 34 shows other effect of diluted acids at different temperatures.

CORE REMOVAL FROM CASTING IN DILUTE ACETIC AND CITRIC ACID (1:1)												
CORE FIRED TEMPERATURE	200 °C			400 °C			600 °C			800 °C		
	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	O	x	x	O	x	x	O	x	x
2A	x	✓	✓	O	x	x	O	x	x	O	x	x
3A	x	✓	✓	O	x	x	O	x	x	O	x	x
4A	x	✓	✓	O	x	x	O	x	x	O	x	x
1B	x	✓	✓	O	✓	✓	O	✓	✓	O	✓	✓
2B	x	✓	✓	O	✓	✓	O	✓	✓	O	x	✓
3B	x	✓	✓	O	x	x	O	x	x	O	x	x
4B	x	✓	✓	O	x	x	O	x	x	O	x	x
2D	x	✓	✓	O	x	x	O	x	x	O	x	x
1E	x	✓	✓	O	x	x	O	x	x	O	x	x
2E	x	✓	✓	O	x	x	O	x	x	O	x	x

O - No effect on core removal

x- Some effect on core removal

✓- Core completely collapses.

Table 34. Effect of dilute acetic and citric acid (1:1)

4.5 CORE COMPOSITION [5] RESULTS

A twenty second dry mix (without water/binder) and another twenty second mean slurry working life after mixing (with water/binder) is adopted for all core trials.

Figure 65 Mean setting time for core trials

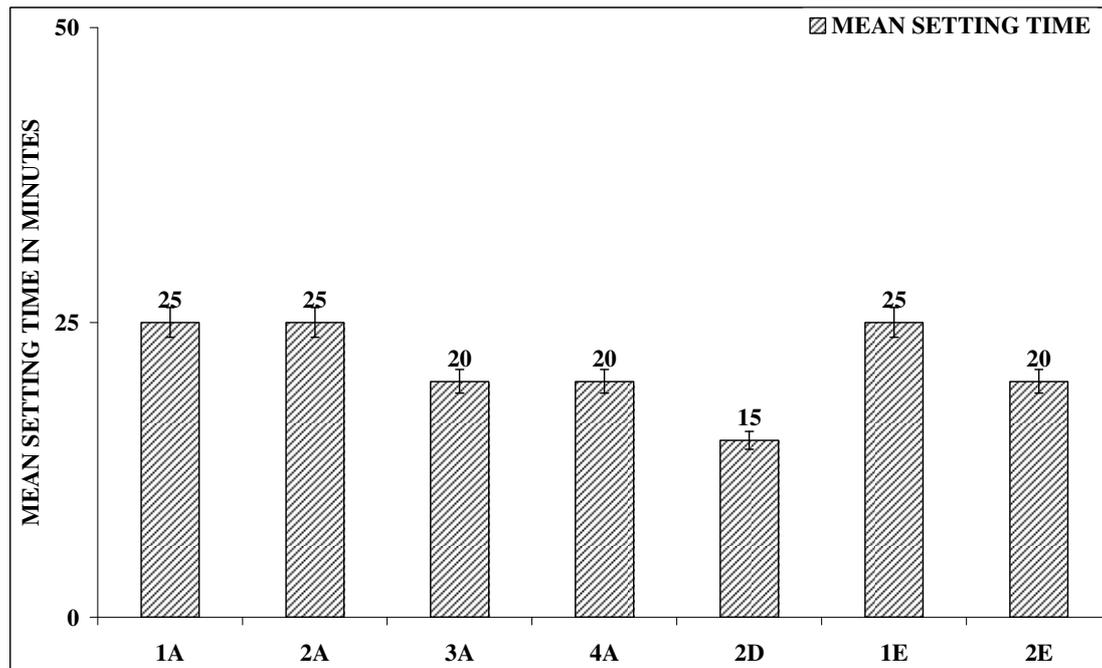


Figure 65. Mean setting time of core trials

Batches of 25 cores were made for each core trial. Batch was left to dry out for twenty-four hours at room temperature in an open box to remove any excess moisture content in cores. The cores were fired to 600 and 800 °C. After the firing process, they were left to cool to room temperature for two hours.

4.5.1 CORE COMPOSITION [5] MEAN DIMENSION CHANGES

The core trial batch dimensions (diameter and length) were recorded before and after firing the cores. Figures 66 to 68 show mean percentage length contraction at different fired temperatures.

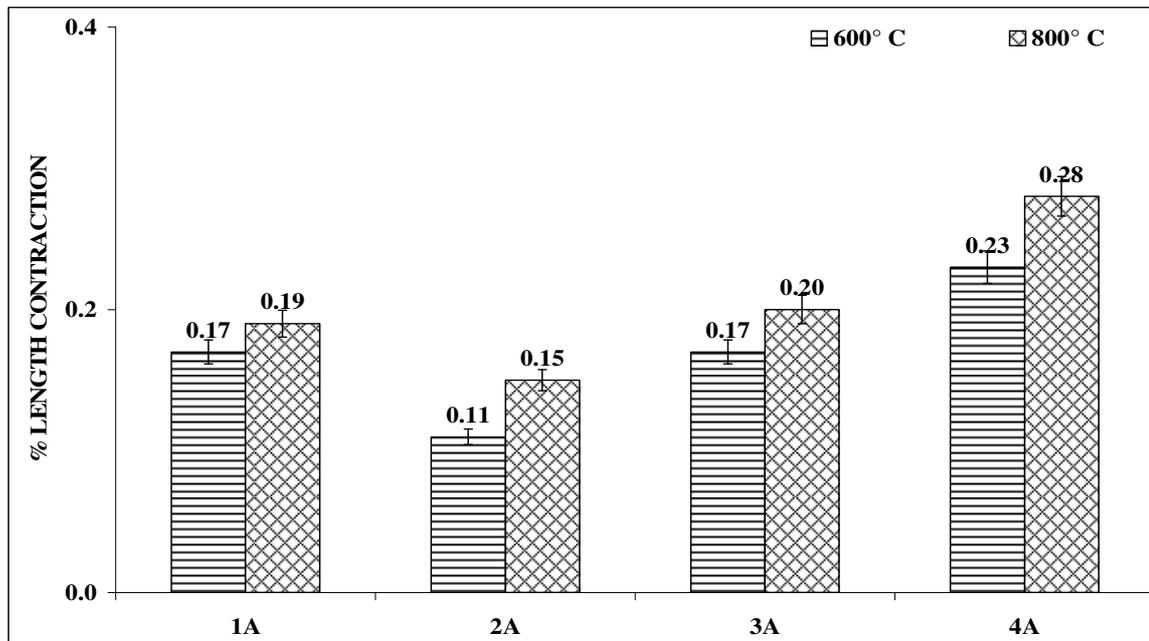


Figure 66. Mean percentage length contraction of Trial A at different fired temperatures

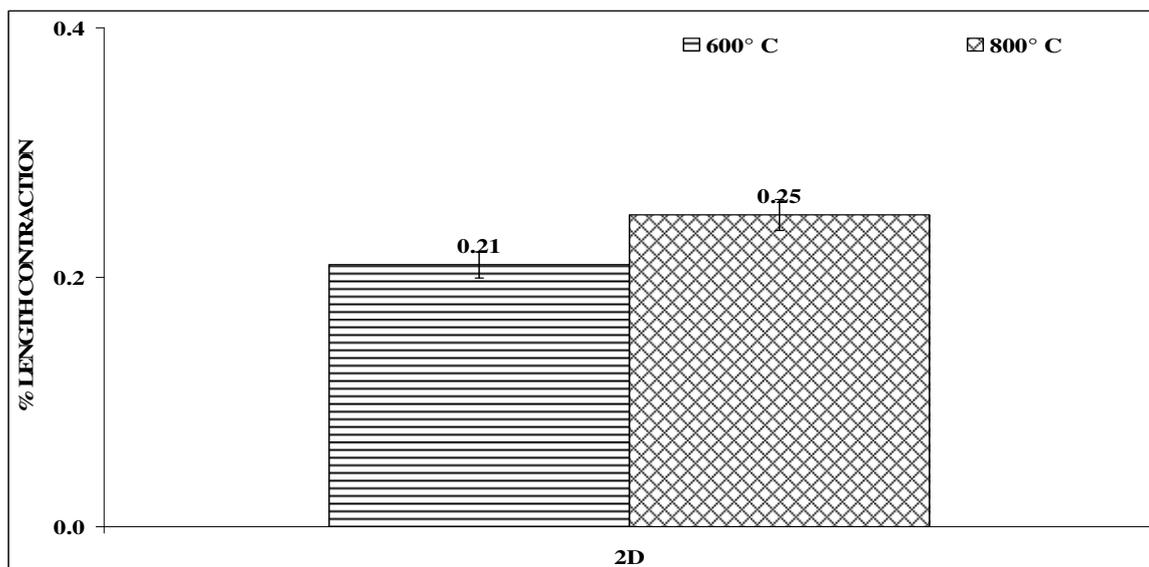


Figure 67. Mean percentage length contraction of Trial 2D at different fired temperatures

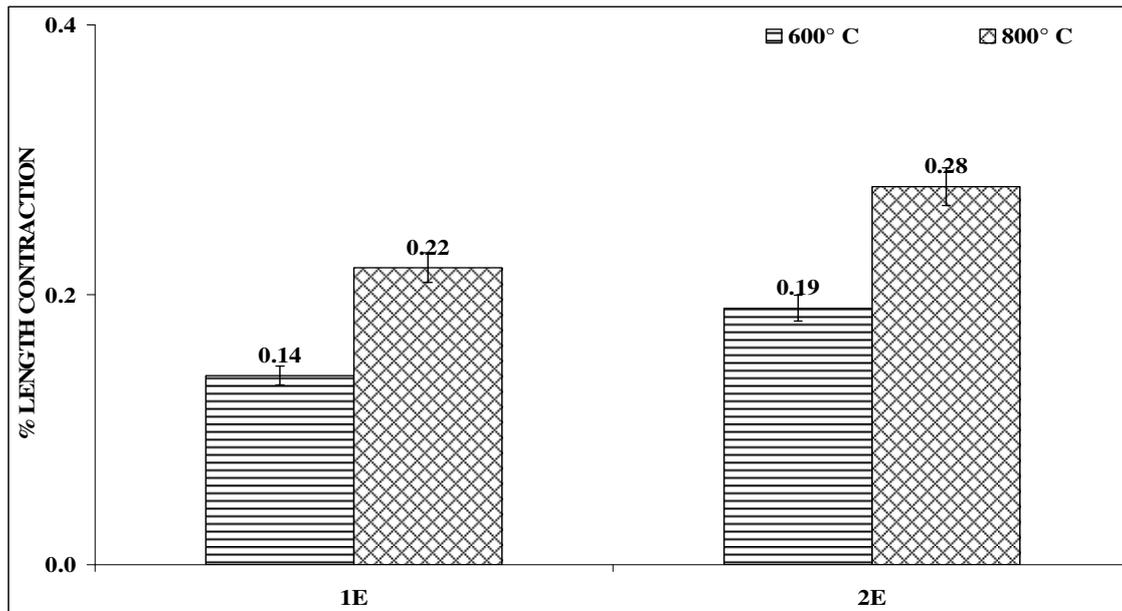


Figure 68. Mean percentage length contraction of Trial E at different fired temperatures

Figures 69 to 71 shows the mean percentage of diameter shrinkage at different fired temperatures

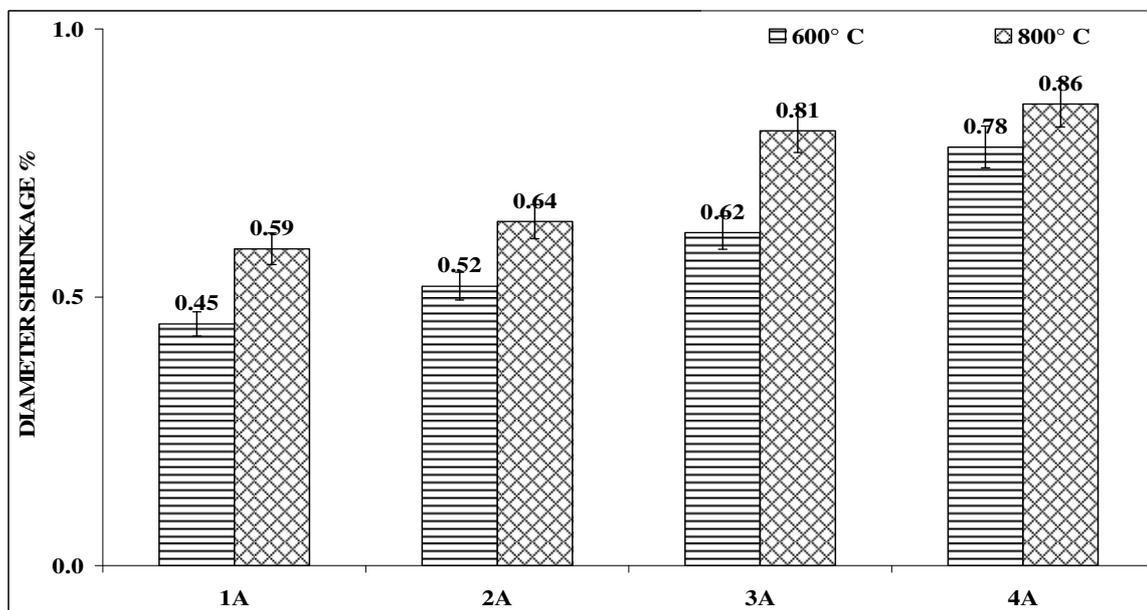


Figure 69. Mean diameter shrinkage percentage of Trial A at different fired temperatures

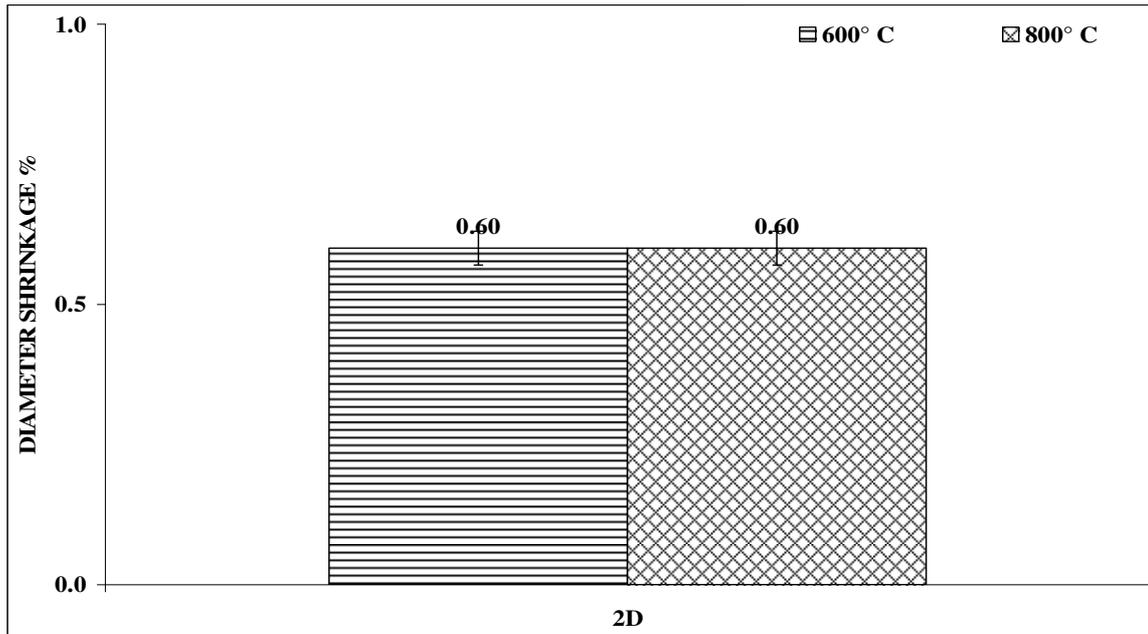


Figure 70. Mean diameter shrinkage percentage of Trial 2D at different fired temperatures

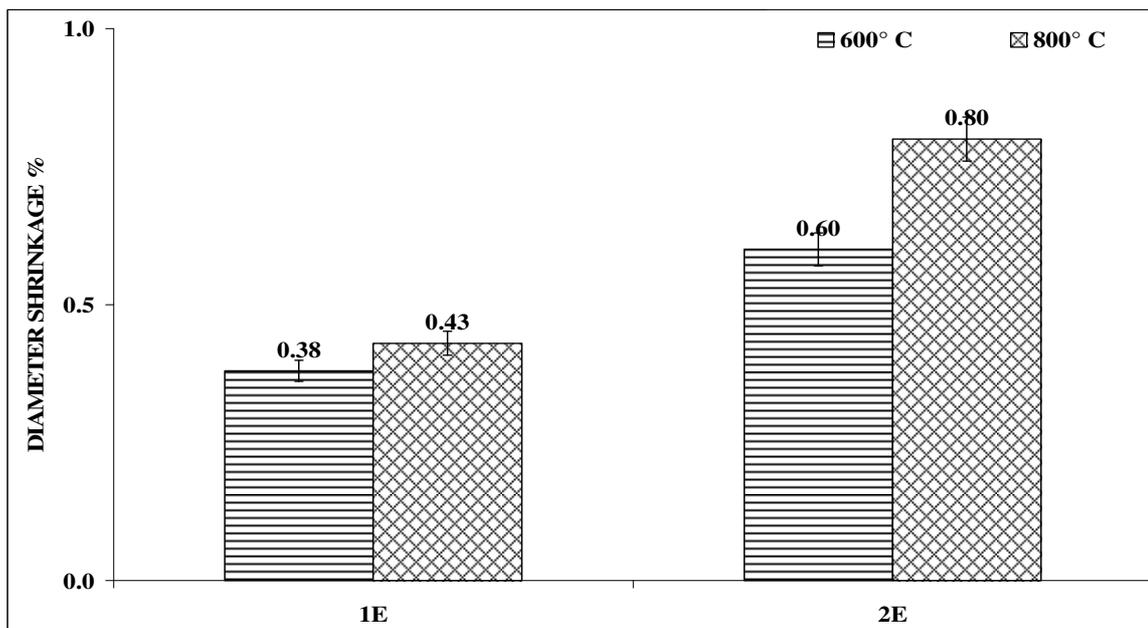


Figure 71. Mean diameter shrinkage percentage of Trial E at different fired temperatures

4.5.2 CORE COMPOSITION [5], MEAN MOR

The mean results of 25 samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 72 to 74.

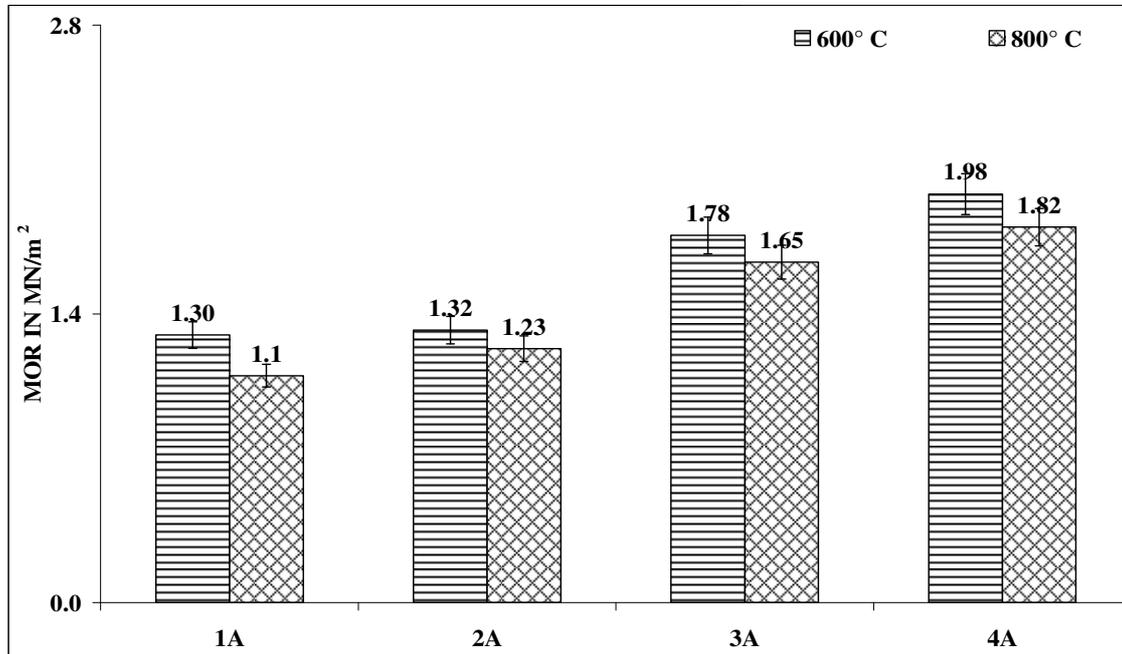


Figure 72. Mean MOR of Trial A at different fired temperatures

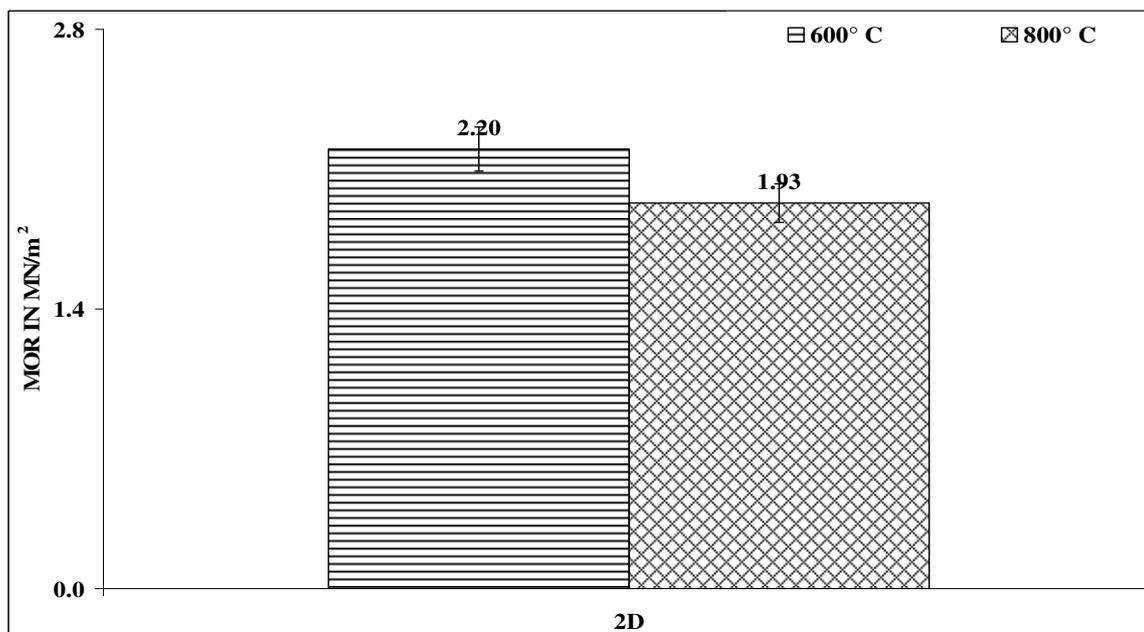


Figure 73. Mean MOR of Trial 2D at different fired temperatures

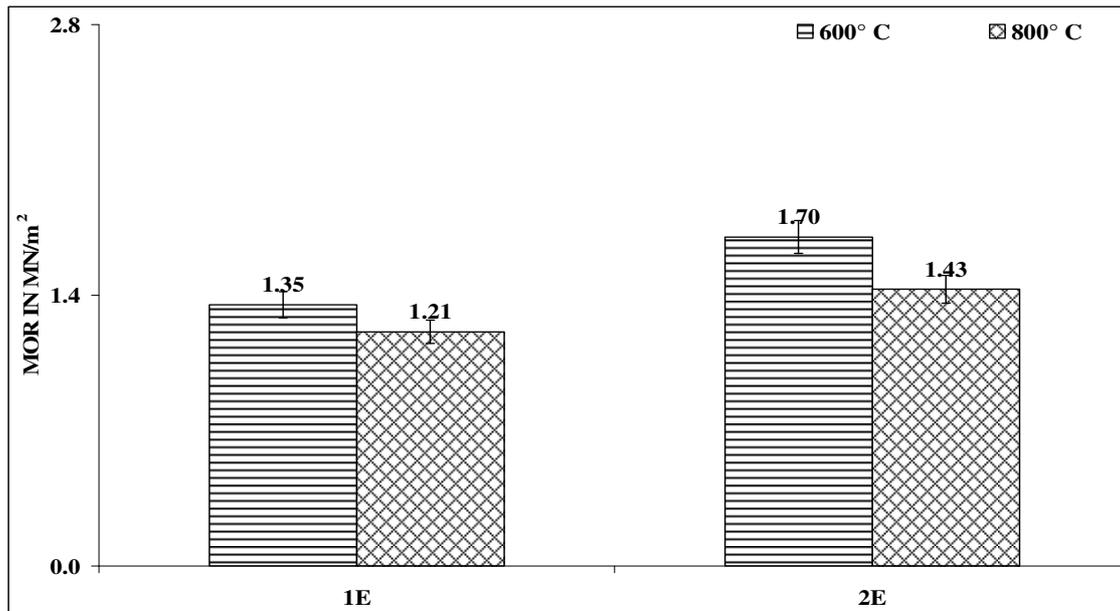


Figure 74. Mean MOR of Trial E at different fired temperatures

4.5.3 K_{IC} AND CRITICAL DEFECT VALUE

The fracture strength (K_{IC}) and critical defect is calculated using equations 10 and 11 derived in section 2.5.5. Tables 35 to 38 show the calculation of the K_{IC} and critical defect size at different fired temperatures

600 °C(K_{IC})						
TRIAL	D(m)	a(m)	a/D	σ (MN/m ²)	Y	K_{IC} (MN m ^{-3/2})
1A	0.0235	0.0062	0.26	1.2	0.6161	0.10
2A	0.0238	0.0061	0.26	1.26	0.6124	0.11
3A	0.0245	0.0061	0.25	1.68	0.6090	0.14
4A	0.0243	0.0063	0.26	1.84	0.6138	0.16
2D	0.0239	0.0049	0.21	2.10	0.5932	0.15
1E	0.024	0.0067	0.28	1.20	0.6245	0.11
2E	0.0238	0.006	0.25	1.59	0.6104	0.13

Table 35. Calculation of the K_{IC} for core trials fired at 600 °C

800 °C(K_{IC})						
TRIAL	D(m)	a(m)	a/D	σ (MN/m²)	Y	K_{IC} (MN m^{-3/2})
1A	0.0236	0.0057	0.24	0.98	0.6057	0.08
2A	0.0238	0.0064	0.27	1.10	0.6188	0.10
3A	0.024	0.0068	0.28	1.50	0.6270	0.14
4A	0.024	0.0066	0.28	1.60	0.6221	0.14
2D	0.0235	0.0053	0.23	1.78	0.5995	0.14
1E	0.0234	0.0075	0.32	1.10	0.6519	0.11
2E	0.024	0.0075	0.31	1.20	0.6461	0.12

Table 36. Calculation of the K_{IC} for core trials fired at 800 °C

600 °C-CRITICAL DEFECT SIZE(a_c)					
TRIAL	Y	K_{IC} (MN m^{-3/2})	σ (MN/m²)	a_c (m)	a_c (mm)
1A	0.6161	0.1032	1.30	0.0008	0.77
2A	0.6124	0.1066	1.32	0.0008	0.78
3A	0.6090	0.1420	1.78	0.0008	0.75
4A	0.6138	0.1593	1.98	0.0008	0.78
2D	0.5932	0.1546	2.20	0.0006	0.55
1E	0.6245	0.1087	1.35	0.0008	0.81
2E	0.6104	0.1333	1.70	0.0007	0.73

Table 37. Calculation of the critical defect size at 600 °C

800 °C-CRITICAL DEFECT SIZE					
TRIAL	Y	K_{IC} (MN m^{-3/2})	σ (MN/m²)	a_c (m)	a_c (mm)
1A	0.6057	0.0794	1.1	0.0006	0.61
2A	0.6188	0.0965	1.23	0.0008	0.75
3A	0.6270	0.1375	1.65	0.0009	0.87
4A	0.6221	0.1433	1.82	0.0008	0.76
2D	0.5995	0.1377	1.93	0.0006	0.58
1E	0.6519	0.1101	1.21	0.0011	1.12
2E	0.6461	0.1190	1.43	0.0009	0.92

Table 38. Calculation of the critical defect size at 800 °C

4.5.4 CORE REMOVAL

The assessment of removal was visual followed by probing with a sharp blade. A single casting was carried out for each trial. Table 39 shows the effect of diluted acids at different temperatures.

CORE REMOVAL FROM CASTING IN DILUTE ACETIC AND CITRIC ACID (1:1)						
CORE FIRED TEMPERATURE	600 °C			800 °C		
TRIAL	1 h	4 h	24 h	1 h	4 h	24 h
1A	x	✓	✓	x	✓	✓
2A	x	✓	✓	x	✓	✓
3A	x	✓	✓	x	✓	✓
4A	x	✓	✓	x	✓	✓
2D	x	✓	✓	x	✓	✓
1E	x	✓	✓	x	✓	✓
2E	x	✓	✓	x	✓	✓

Table 39. Effect of dilute acetic and citric acid (1:1) on core trials

4.5.5 DISTRIBUTION OVERVIEW PLOT, WEIBULL

The Minitab[®] software was used to plot the distribution overview plot. Figures 75 to 78 show the distribution overview plot at different fired temperatures.

X-axis- Data- MOR from each core Trial.

Y-Axis- Weibull Parameter

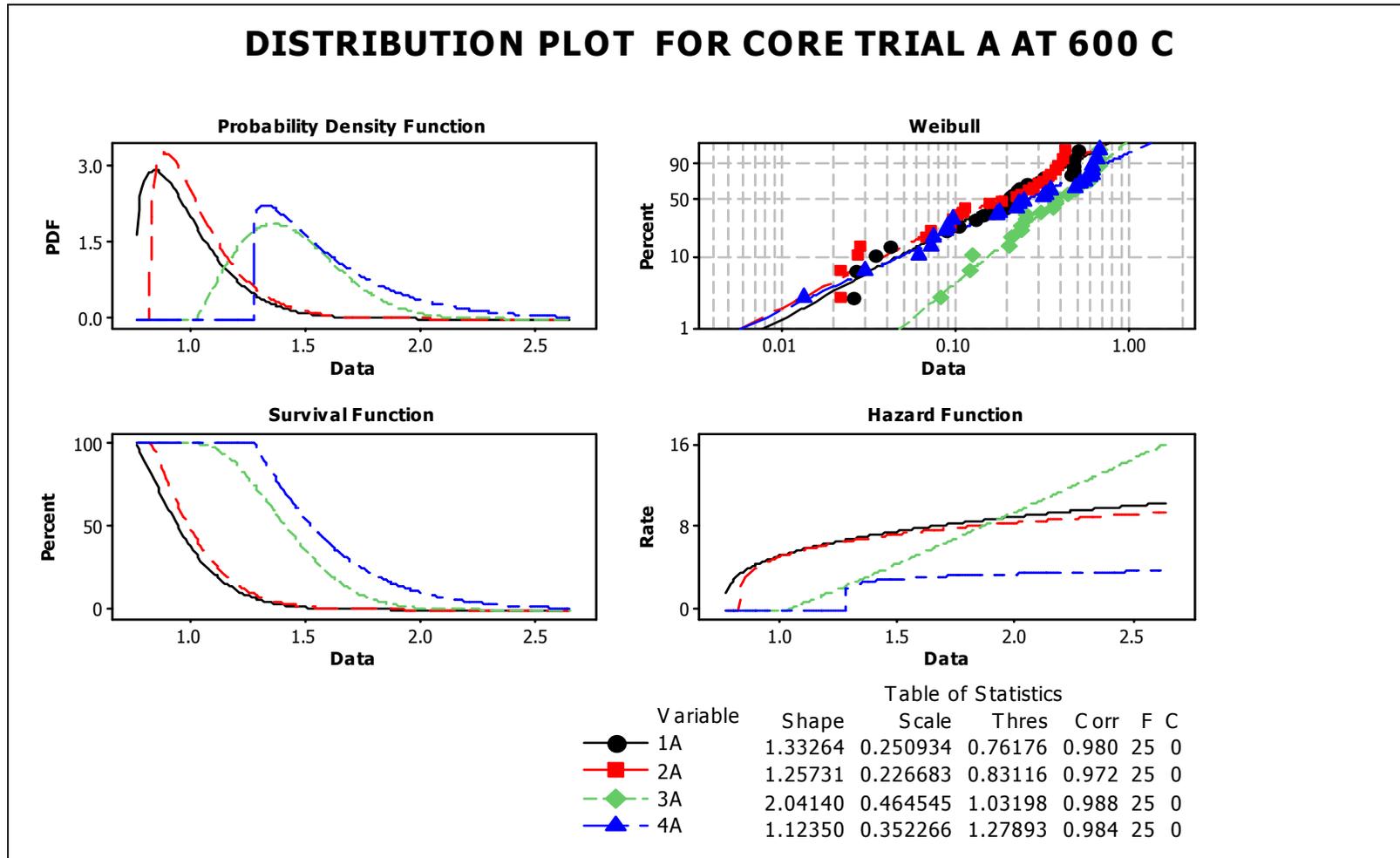


Figure 75. Distribution overview plot for Trial A at 600 °C

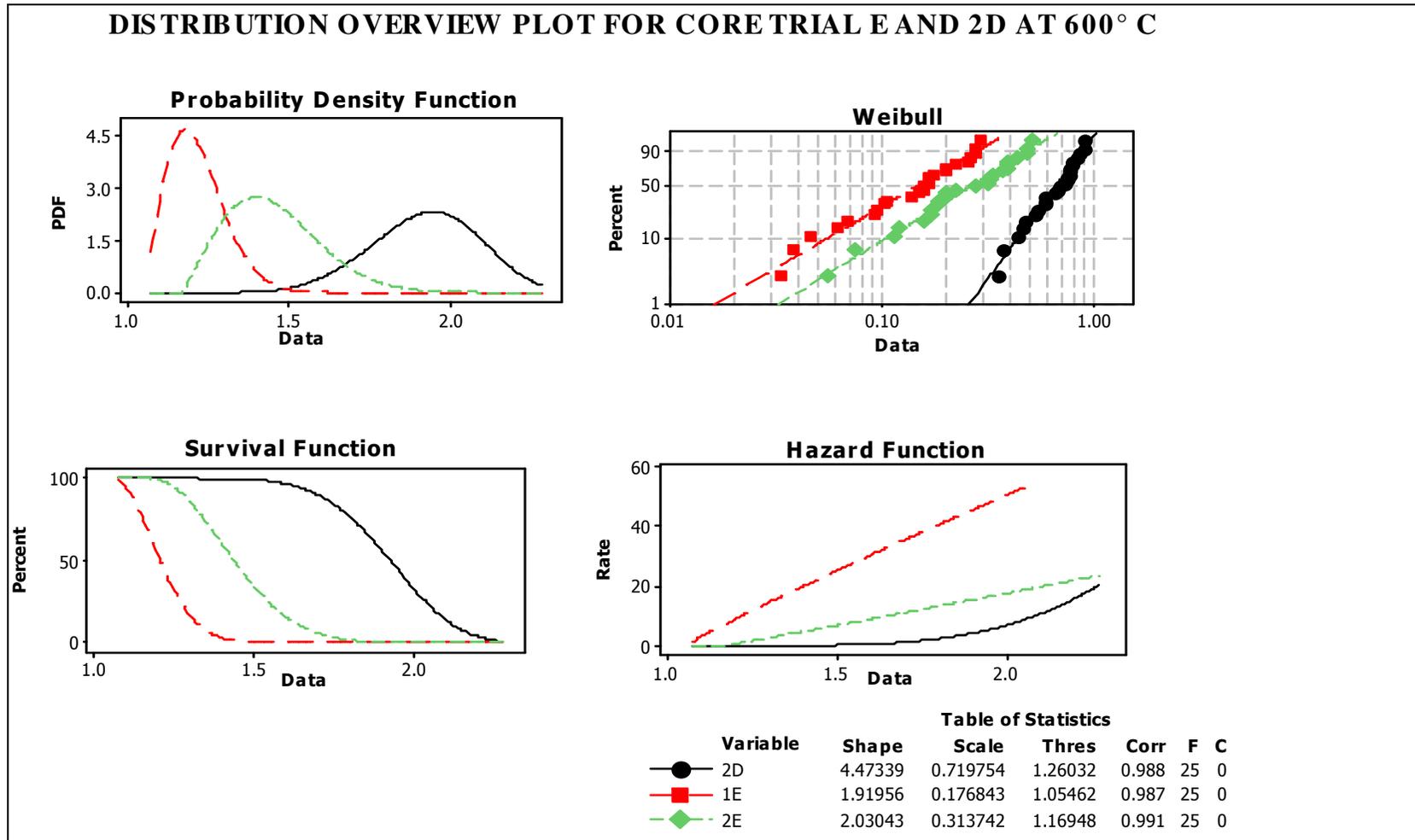


Figure 76. Distribution overview plot for Trial E and 2D at 600 °C

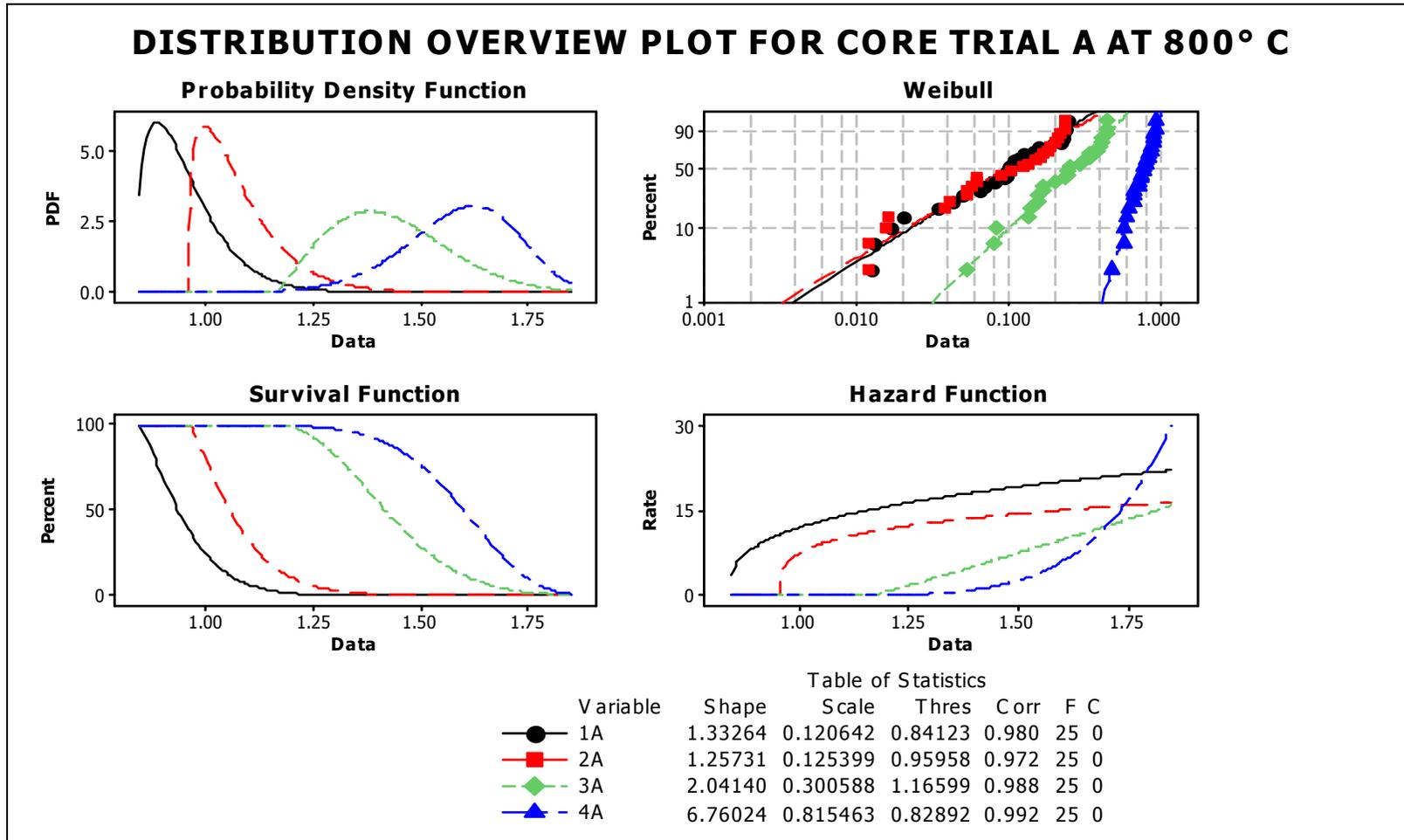


Figure 77. Distribution overview plot for Trial A at 800 ° C

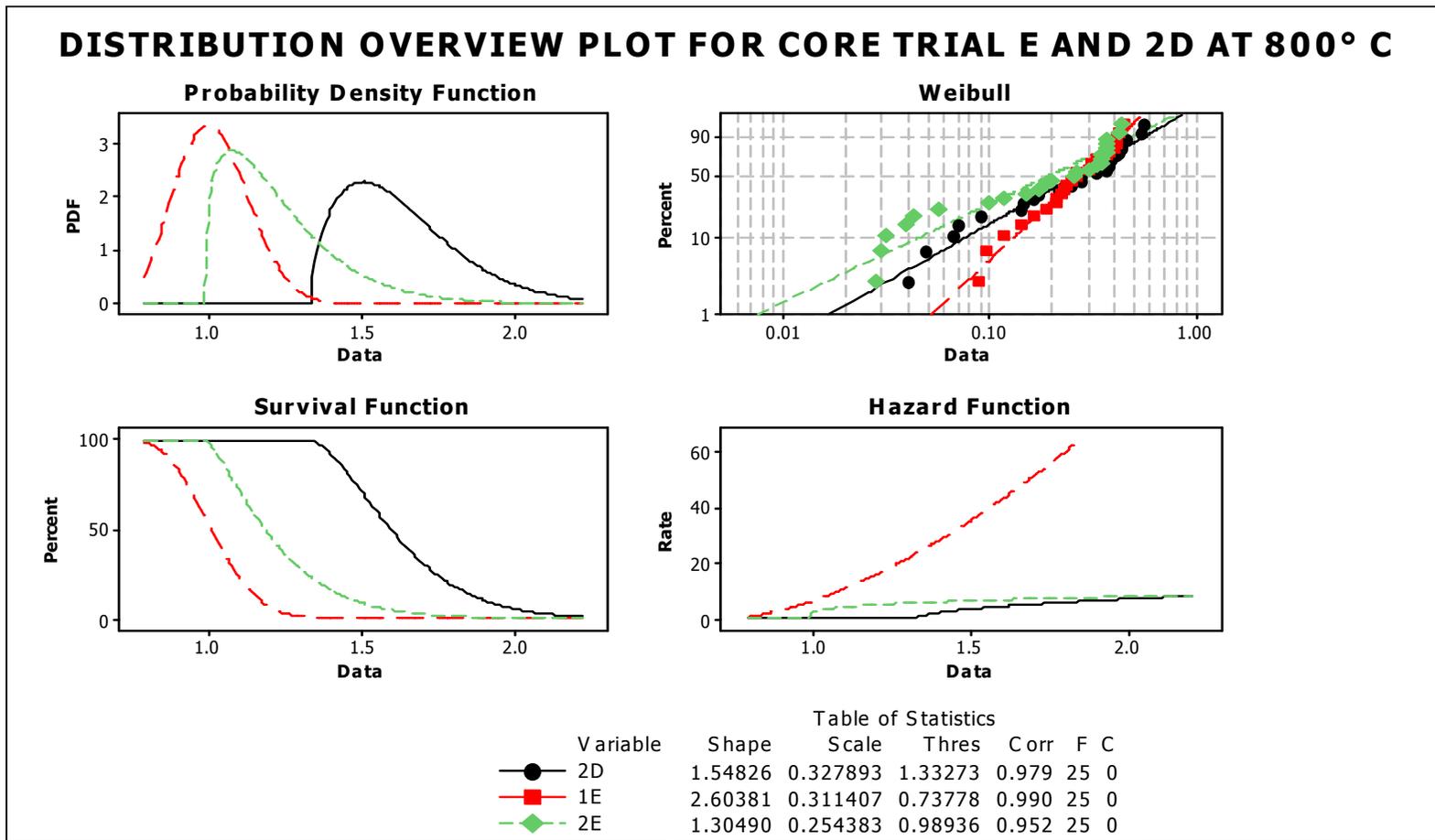


Figure 78. Distribution overview plot for Trial E and 2D at 800 °C

4.6 CORE COMPOSITION [6] AND [7] RESULTS

Batches of ten samples were produced using core trials. A twenty-second dry mix and another twenty-second mean slurry working life after mixing process were adopted for all core trials. Figure 79 is shows in Section 5.5.3(page 171). Figure 80 Mean setting time for core trials.

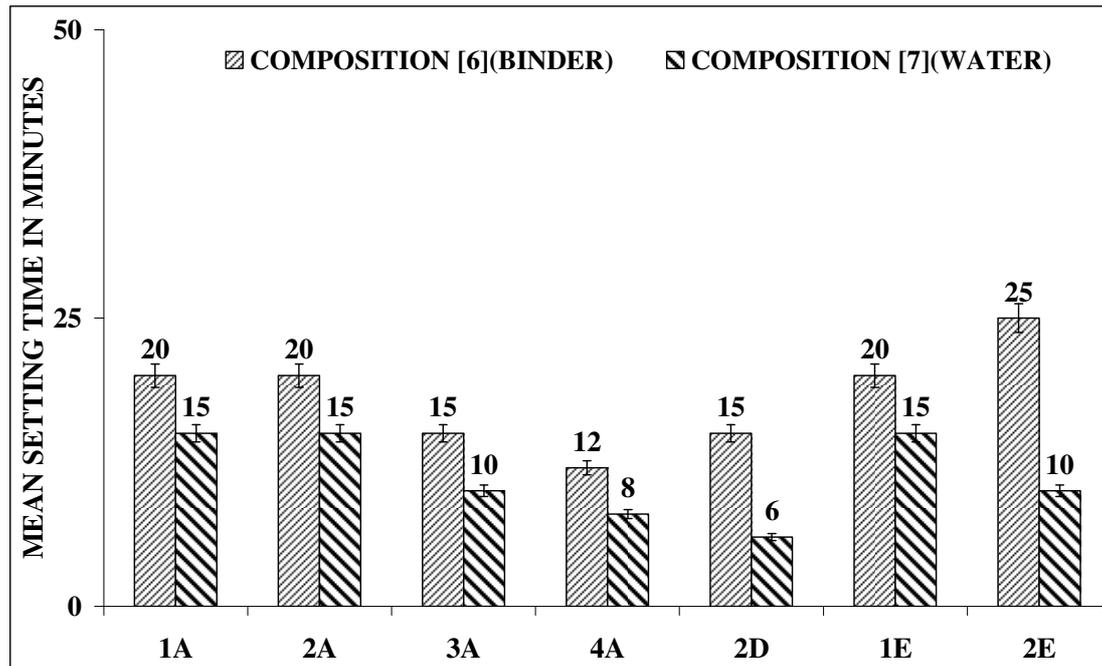


Figure 80. Mean setting time for core trial

4.6.1 CORE COMPOSITION [6] AND [7], MEAN MOR

The mean results of 10 samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 81 and 82.

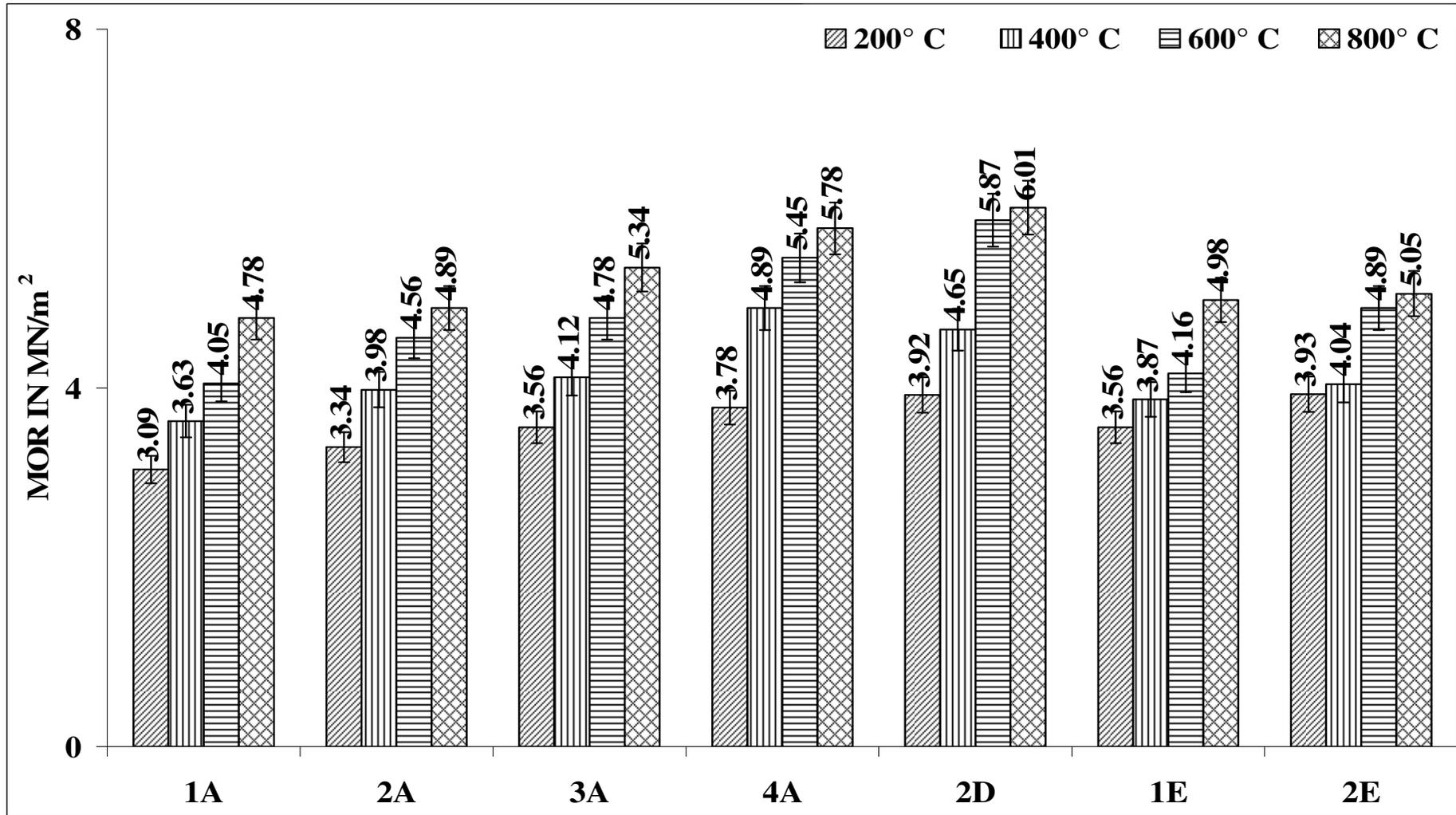


Figure 81. Mean MOR for core composition [6] at different fired temperatures

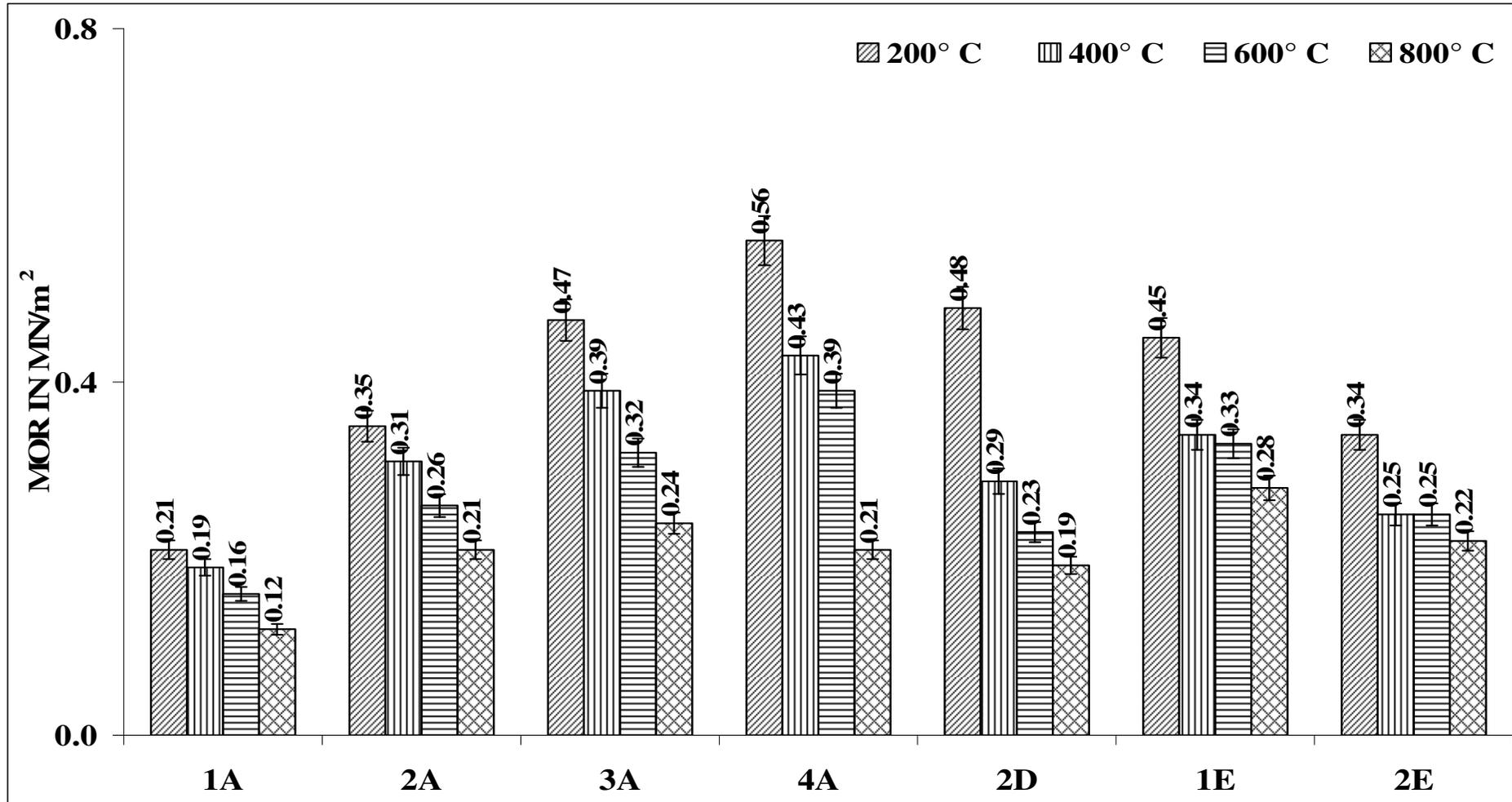


Figure 82. Mean MOR for core composition [7] at different fired temperatures

4.7 CORE COMPOSITIONS [8] AND [9] RESULTS

Batches of five samples were produced using core compositions [8] and [9]. A twenty-second dry mix and another twenty-second with mean slurry working life after mixing process are adopted for all core trails. Figure 83 Mean setting time for core trials

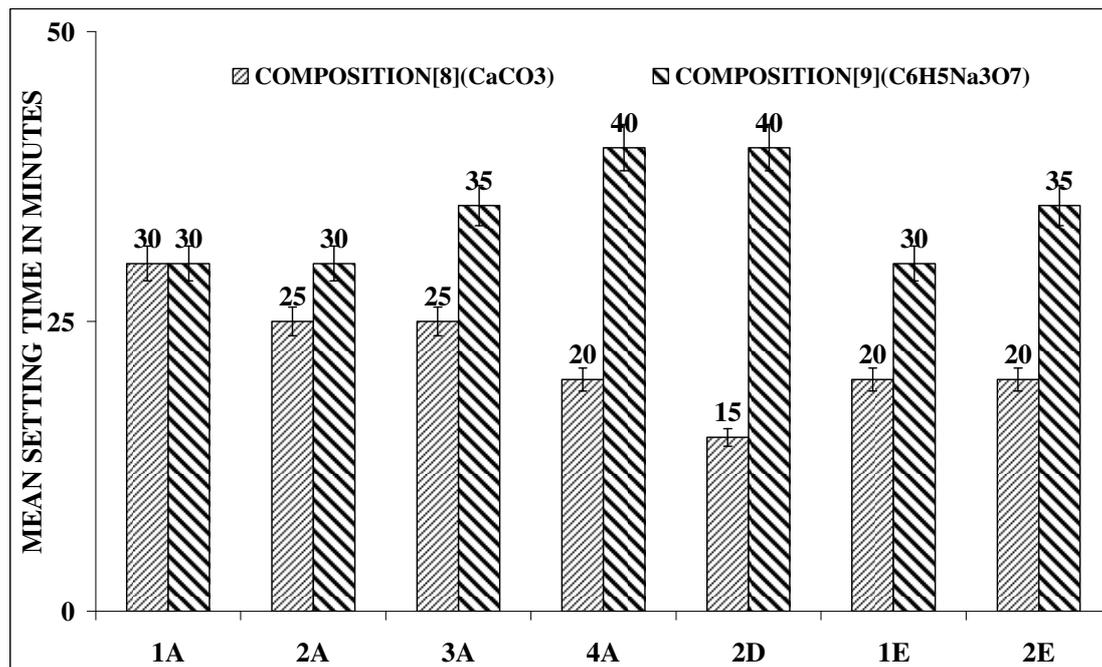


Figure. 83 Mean setting time for core composition [8] and [9]

4.7.1 CORE COMPOSITIONS [8] AND [9], MEAN MOR

The mean results of five samples for each core trial tested using three-point bend testing (MOR) are shown in Figures 84 and 85.

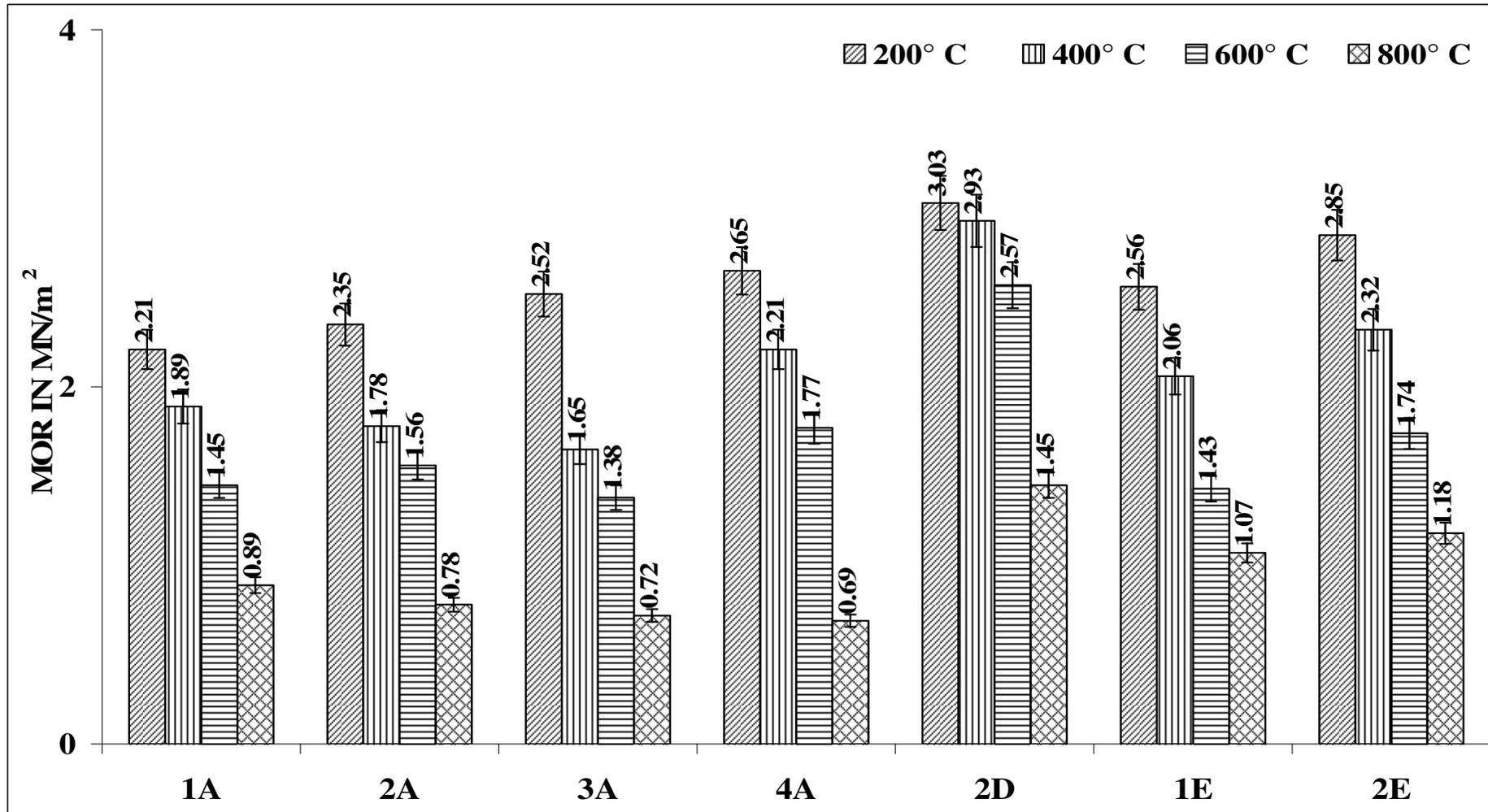


Figure 84. Mean MOR for core composition [8] at different fired temperatures

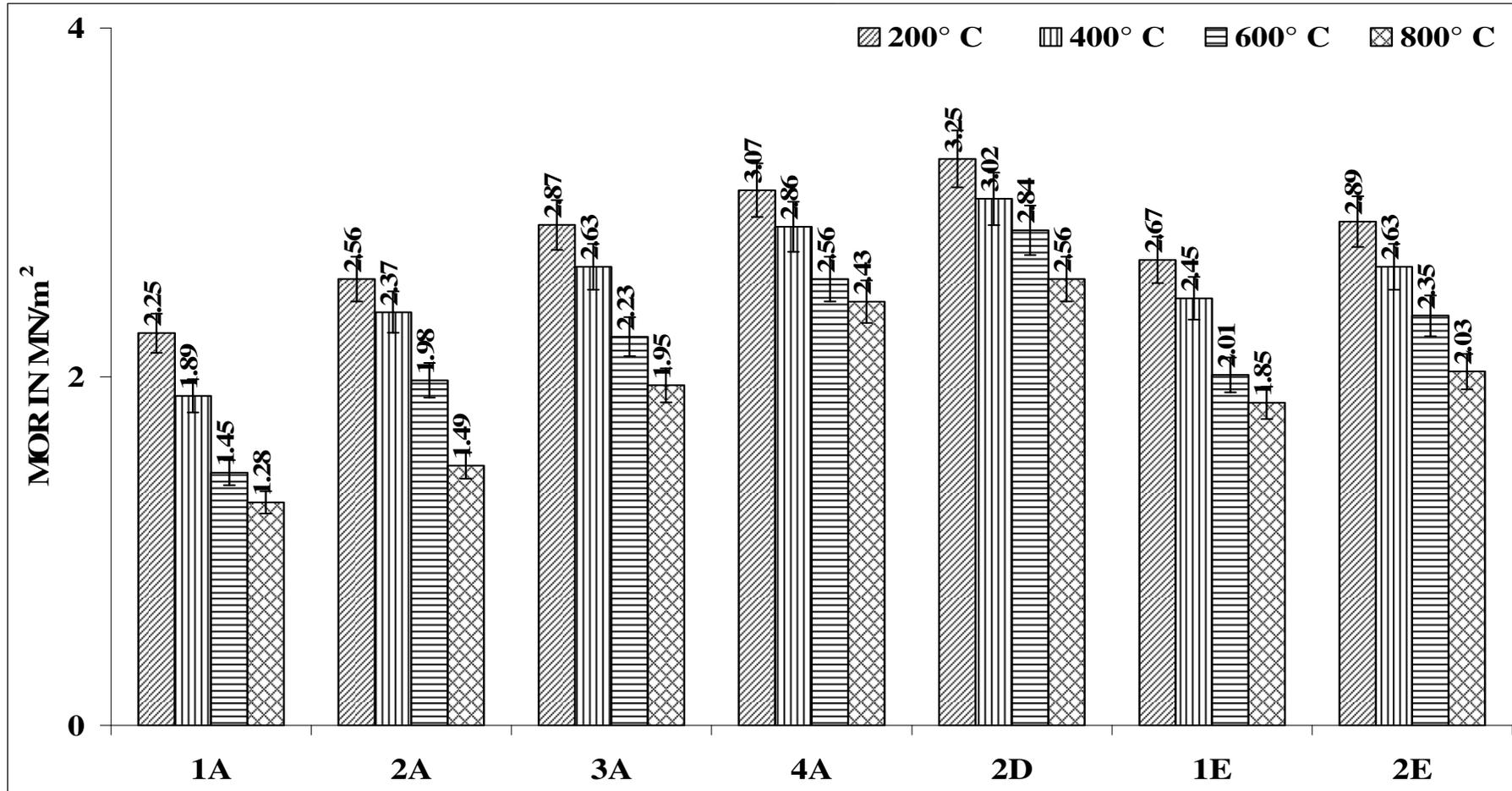


Figure 85. Mean MOR for core composition [9] at different fired temperatures

5.0 DISCUSSION

This chapter considers the results presented from core manufacturing, testing, casting, and core removal from the cast part. The suitability of the manufacturing process utilised for producing experimental cores and the subsequent testing methodology is discussed. The different core trials are then sequentially evaluated in terms of composition, manufacturing, mechanical testing, suitability for casting and core removal. Generic aspects of the programme are then evaluated.

5.1 EVALUATION OF EXPERIMENTAL TECHNIQUE

Readily available softwoods were used to make the core boxes for cheapness and ease of machining. Wear on the cavity surfaces of the core box resulted in some rough surfaces on the cores, (which were used in mechanical testing). In commercial use, a more wear resistant material (e.g. steel) might be used. In the case of this study, the surfaces were occasionally repainted as required.

The use of a hand held plaster mixer to mix the core compositions in the dry state was effective in giving a uniform blend in just a few seconds. It is reasonable to assume that effective mixing of commercial quantities could be achieved in a comparable or acceptable period of time. Mixer blades were cleaned everyday to remove rust. Commercially, a stainless steel paddle might be used.

The addition of the premixed binder/water caused the setting process to commence. In the case of several compositions, setting commenced instantaneously (three seconds as measured). It was possible to manufacture experimental cores within this time but

commercial use would therefore be precluded. This influenced the further programme of experimental work.

Cores were left to dry at room temperature for twenty-four hours, to allow removal of maximum moisture and possibility of subsequent thermal shock effect⁽⁴²⁾. All cores produced, none showed any evidence of post-fire cracking using this procedure, which is practised by some commercial manufacturers⁽¹⁴⁾. Vacuum drying did not seem to give any significant advantage, except perhaps minimally in time, but at extra cost.

The weight loss measurements were carried out using a three fraction scientific scale. The scale was recalibrated every 100 readings according to manufacturer (Fisher). No adjustment was necessary on any occasion. The diameter and length were measured using micrometer and vernier callipers respectively. The muffle furnace used for firing was controlled by a ElectroxTM temperature controller. Furnace control was to within +/- 10 °C. This is probably better than might be achieved throughout the hot zone of a commercial kiln at 600 °C and lower. It is not thought likely that temperature disparity a little greater than this would have significant effect on the product. Some variations of temperature may be recorded when loading and unloading cores from the furnace; these would have negligible effect as long as the firing time was significantly greater than the furnace temperature recovery time. The cooling process was carried out in the muffle furnace to room temperature for two hours.

All cores had sufficient strength to be handled through processing and then to testing. Some cores exhibited anomalous displacement as the loads were initially applied due to micro-roughness of the surface. Fracture toughness test piece preparation, although requiring manual machining of a brittle material, did not cause any premature fracture of test pieces and all were successfully notched and tested. Such degree of robustness is essential for commercial processing.

5.1.1 CORE COMPOSITION [1] DISCUSSION

Core Drying. Some initial experimental work was undertaken in core composition [1] of development work to establish some basic parameters. One of these was the extended vacuum drying of core materials. Cores left for twenty-four hours in vacuum showed enhanced moisture loss over those left in atmosphere as expected. The core trials however showed that after two hours firing at different temperatures, all the moisture was removed from the plaster and left a dry core ready for casting. Figures 19 and 20 show the weight loss percentage in different core trials at 200 and 400 °C. The core trials were loaded and unloaded from the muffle furnace at respective firing temperatures. This may have caused some of the core recorded weight loss at four hours. The cores lost least weight after four hours. It is suggesting that two hours of firing cycle is sufficient to remove water crystallisation.

The mixing processes with (binder/water) was very quick. The mean slurry working life is the time of slurry set to in their cup after mixing are carried out.

5.1.2 COMPOSITION [1] PROPERTIES AND REMOVAL

Trial A, The allowable slurry working life, before stiffening of the mix occurs, is significantly reduced as the plaster content increases. Trial compositions 2A and 3A had six seconds as working life whereas the working life for 1A and 4A was eight and four seconds respectively. This is due to the higher magnesium oxide (1%) reaction with calcium sulphate. Magnesium oxide composition at a level of 1% is too high, for any of the compositions. None of these times is long enough in a commercial ⁽⁷⁶⁾ environment, or with large batch quantities.

Trial 1A core required care in manual handling, as it was weak when removed from the core boxes. Trials 2A and 3A were sufficiently robust and 4A even more so. The less percentage of plaster, the longer it took for the cores to set hard. The mean setting time in core box for 1A was thirty minutes, for 2A and 3A, twenty-five minutes and for 4A twenty minutes. The longer times might not be adequate for a commercial application. Trial A shows that it must be decided whether workability or strength is more important. The precise consistency to use will depend on the individual commercial application.

The strength of ceramic cores is related to fired temperature. Figure 27 shows the mean MOR at different percentage of plaster added on Trial A. The mean MOR is reduced as the temperature is increased due to the plaster losing water, evaporation of water crystallisation and all other compounds losing bonds. Firing at 200 °C gave higher Modulus of Rupture (MOR) due to presence of carbonates (like Li_2CO_3), which are thermally stable on moderate heating.

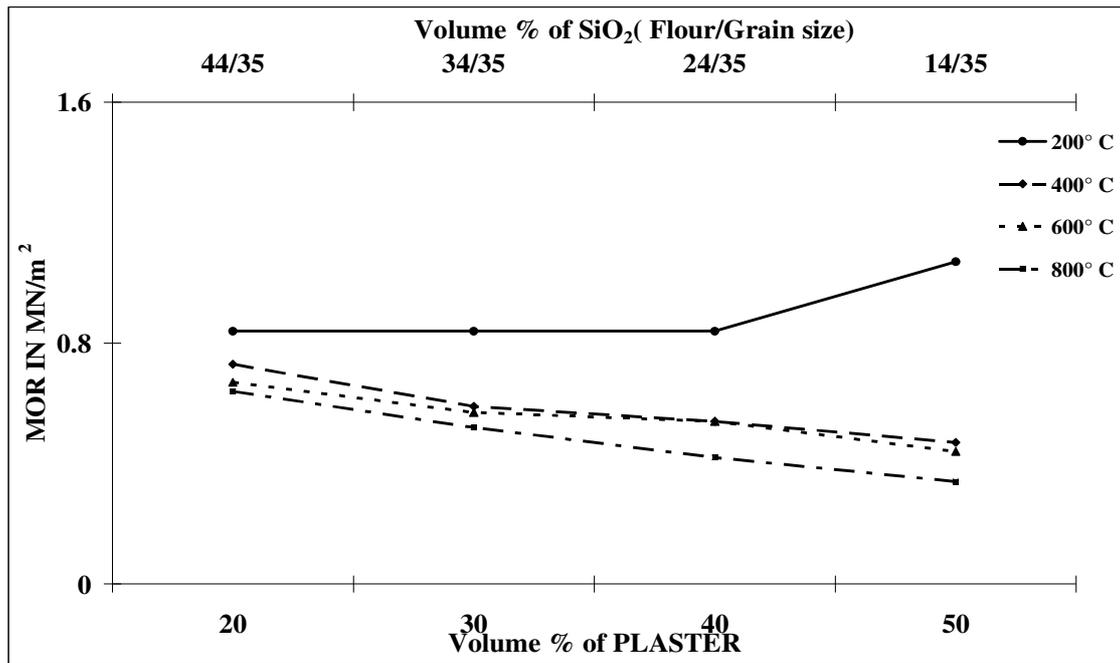


Figure 27. Mean MOR in Trial A at different fired temperatures

At 200 °C, dilute nitric acid (1:10) had more effect on trial cores 1A and 4A than any of the other trials. After four hours, the core was soft due to dilute nitric acid slowly attacking the bonds of complex compounds. The acid had an effect on 1A, which was fired to 400 and 600 °C. Within the first hour, the core was soft inside the cast part. Manual removal was easily achieved. After leaving for twenty-four hours, the core disintegrated. After close examination however, core materials were seen remaining on the internal faces of the cast part. Jet washing would still be required to remove the residue.

The surface finish of the cast part internals was rough indicating metal penetration. At different temperatures, the acid did have effect on 2A and 3A (that contained a higher proportion of plaster). Trial 4A however was attacked within twenty-four hours. The core Trial A suggests that with plaster percentage (30 to 40%), that a core fired at an intermediate temperature (400 , 600 and 800 °C) was susceptible to break down.

In Trial B, the effects of change in concentration of binder/water on core trials were analysed. Trial 1B (higher water percentage) and 4B (higher binder percentage) had a very significant effect on the slurry working life. 1B gave shorter working life than 4B, due to oxygen compounds in water quickly reacting with calcium oxide compounds. The material in Trial 1B set quickly if it was not poured immediately. It is suggesting that MgO with water gives an instant mix, resulting in short slurry working life. It has a large percentage of water and suffers from a commercially unsustainable pouring time of less than five seconds. The higher proportions of binder give a highly viscous mix (and longer slurry working life). It is suggested that Ludox AM takes longer to bond with oxides of other compounds. On average, Trials 2B and 3B had the same slurry-working life time even though binder/water concentration varied.

Trial 1B and 4B had quick setting times in the core mould box. The material in Trial 1B was brittle and weak, that in 4B being strong with a smooth surface when removed from the mould. Trials 1B was very 'green' (too much moisture and rough on the surface). Trials 3B and 4B had the same times for setting.

Figure 22 shows the mean MOR values of Trial B core compositions. The material in Trial 4B (at any temperature) has higher strength than other those in trials due to higher percentage of binder. When binder (Ludox AM) was heated at higher temperatures, dried particles of binder develop strong adhesive ⁽³⁰⁾ bonds. The products from Trial 1B (at all temperatures) recorded lower mean MOR since the evaporation of the water has caused the disruption of bonds, making it brittle and weak. Trial 3B at different temperatures gave higher mean MOR than Trial 2B due to

higher percentage of binder. The small changes in binder concentration increased the strength of cores. Were such cores (Trial 1B and 2B) be used in investment casting, the stresses involved in injection moulding of wax patterns would inevitably cause core fracture. The addition of binder concentration can alter the strength required in ceramic cores.

All of the variants of Trial B fired at 400 °C were attacked by diluted nitric acid on the plaster components. Mixed core trials were turning into salt in four hours of dipping in diluted nitric acid since the compounds became unstable. Indeed, within twenty-four hours core trials were turned into slurry. Further mechanical removal was not necessary, as the core had already disintegrated. Cores fired at 600 °C were attacked by diluted acid after 4 hours and cores were soft after 24 hours. At 200 and 800 °C, there was no significant effect at one hour, possibly due to the presence of calcium induced glassy phases. The Trial B, suggest that binder/water concentration can be used to vary the strength, this would be crucial in any commercial industrial application.

Trial C, Trial 3C contained a significant (in core making terms) percentage of magnesium oxide. The material in Trial 3C had only three seconds of slurry working life. It became hard very quickly even during the mixing process. This rendered pouring difficult as it began to set even before pouring into the core mould box. Trial 1C gave a longer slurry working life to mix and pour since there was no magnesium oxide present. Trail 2C gave five seconds of slurry working life, similar to Trial 2A. The trial was conducted to see changes in setting of mixed slurry in core trials with different percentage of magnesium oxide.

Trial 1C cores were weak and brittle when removed from the mould. Even after twenty-four hours, cores were very wet due to bonds not completely forming and cores sometimes broke in the removal process from the mould. As with all other core trials, the core surface had voids and splits. The material in Trial 2C was sufficiently robust, once settled; the material in Trial 3C set hard more quickly. The core outer surface had small pores when removed from the core mould box. This may be due to air trapped in the mixed slurries which were setting quick.

When fired at different temperatures, product from Trial 3C had cracks on the surface. These cracks were due to internal pores contracting and the external surface expanding in the firing cycle. Such cracking may lead to core failure in any industrial process, such as wax pattern injection or manual handling. Material from Trial 1C and 2C had a rough surface; no cracks were found, apart from usual splits and voids visible on the surface.

Figure 23 shows that material from Trial 2C has similar mean MOR ($\pm 10\%$) as that from Trial 2A, suggesting the formation of the same compound. Since producing a core was difficult for Trial 3C due to higher addition of magnesium oxide (2%), MORs (at all temperatures) were very low. The core is not suitable for use in any applications as the setting time is too quick, not to mention the presence of surface cracks.

Whether the cores were fired at 200 °C, 400 °C, 600 °C and 800 °C, diluted nitric acid had no effect on the cores, only the former softening after twenty-four hours. This is due to cores may have formed stable complex bonds. The surface finish of the cast

part was smooth despite the core surface appearing rough. Trials C suggest that magnesium oxide is influencing the setting of the mixed slurry.

In Trial D, core trials were prepared to examine the effect of calcium silicate. The slurry working life of material in Trial 1D was same as those in Trials 2A and 2C. The material in Trial 2D gave a longer working life since there was greater percentage of binder, which was an improvement, though not sufficient for industrial conditions. After removal from the wooden mould box, the cores had no visual indication of pores on the outer surface. The core trials could be readily handled without failure.

Figure 24 shows the mean MOR of Trial D at different fired temperatures. The product from Trial 1D has a lower mean MOR (at all fired temperatures) than that from Trial 2D since binder percentages are higher. Trial 1D gave very similar mean MOR as Trial 2A. The addition of calcium silicate in Trial 2D forms more complex and stable compounds with other core materials that influence the higher mean MOR.

Diluted nitric acid had no effect on product from Trial 1D at 200 °C. At temperatures of 400, 600, and 800 °C, cores were soft when removed using a sharp edge within twenty-four hours. At 400 °C, core from Trial 2D was soft within one hour, the calcium silicate bonds being readily attacked by diluted nitric acid. The core was very soft in the cast part and broke-up in twenty-four hours. At other fired temperatures, Trial 2D cores were soft after four hours. Trial D suggest that addition of calcium silicate may be increasing the strength and collapsibility property of core trials in diluted nitric acid.

Trial E Special type of plasters were used to prepare these core trials. The Crystacal LX Plaster (CLX) was hard even before the mixing process could be completed. The addition of binder/water made it hard immediately. CLX is indeed designed to be used where very quick setting⁽²⁷⁾ is required. Several attempts were made by changing the different volume proportions but without success, after which attempts to use plaster CLX were discontinued. Trials 1E (Fine Casting Plaster) and 2E (Crystacal R), gave longer slurry working life using the standard core make up. The setting time for Trials 1E and 2E were the same even though Trial 2E used high purity gypsum (hard plaster) produced from naturally occurring⁽²⁷⁾ gypsum.

Trials 1E and 2E cores were hard, and had very smooth surfaces when removed from the wooden core box. The cores had no voids or pores on the surface. The cores were strongly set without any cracks on the surface. The cores had an ideal setting time and easy manual handling influencing the best use in commercial industry.

Figure 25 shows the mean MORs of Trial E at different fired temperatures; product from Trial 2E had higher mean MOR, consequence of the use of high strength hemi-hydrate plaster. Product from Trial 1E had very similar mean MOR compared with plaster (dental plaster) used in other Trials A, B, C and D. The dental plaster and fine casting plaster are beta plasters designed for moderate strength and quick setting.

Dilute nitric acid (1:10) had some effect on all Trial E cores which were fired at different temperatures. At 200 °C, the cores were soft after four hours indicating that the bonds were broken very slowly. At other temperatures, 1E and 2E bonds were broken within four hours losing all chemical properties. Trial E demonstrates that a

change of plaster type can increase the smoothness of the surface, increase the strength and may be leaching properties of the core.

5.1.3 CASTING WITH CORE COMPOSITION [1]

All core trials fired at 200° and 400 °C, released more gas than cores fired at 600 and 800 °C due to extended firing occurring when melted aluminium was poured into the mould cavity(dies). At 200° and 400 °C, the core trial oxides are reacting with molten aluminium (680 °C). The outer surface was dull when left in diluted nitric acids since aluminium is made passive by nitric acid ⁽⁶¹⁾ and this has been attributed to the formation of an impenetrable oxide layer on it surface. Figure 28 shows a core blow defect in the sectioned Al-LM25 after leaching out Trial 1B core fired at 400 °C.



Figure 28. Core blow defect on the sectioned Al-LM25 (core removed)

The core blow occurs by gases released from core before solidification. These have a catastrophic effect on cast parts. In the molten metal pouring process, the metal hits the core surface. Since the feeder is in the centre of the mould (Figure 14), the gases released have no quick escape. This may cause an aluminium casting to fail mechanically since induced gases make aluminium brittle. In sand casting, the molten metal is passed through channels filling the mould. This method is better since the molten metal does not touch the core surface in the filling process. The released gas from core will escape through “air holes” provided in the cope and drag.

5.1.4 CORE MATERIAL DISTRIBUTION

All core trials had a major problem with core diameter and core strength. The wooden core boxes were held vertical during filling as shown in Figure 29. The 30/50 fused silica settled on the bottom of the core box for all cores. The repeated differentiated mixing attempts and the use of horizontal wooden core boxes was not effective, similarly resulting in non-uniform dimensions and a rough surface. Figure 30 shows the 600 °C cores fired for two hours. The colour difference occurs because of the settling of the fused silica 30/50.



Figure 29. Vertical standing wooden core boxes with cores



Figure 30. Cores fired at 600 °C with the 30/50 fused silica settled in the bottom.

5.1.5 REVIEW OF CORE COMPOSITION [1]

The discussion of core composition [1] has suggested changes in all core trials to achieve the aim of the project. The core composition [1] suggested that two-hour firing and two-hour cooling was adequate after twenty-four hours room temperature drying. The core trials have faster reaction with higher percentage addition of water. The composition [1] has also suggested reducing the magnesium oxide percentage to increase the slurry working life. The strength (mean MOR) recorded from the core trials is very moderate and increase in strength is required. The change to a finer particle size of fused silica should give higher strength. To increase the strength, fused silica mesh size was changed -200 size. The other suggestion was to see if fused silica grain size (30/50) could have better distribution with -200 mesh size.

The diluted acids have leached the cores fired at moderate temperatures. Boiling the diluted acid would have given higher leaching rate. The health and safety regulations have limitations on boiling or heating diluted nitric acid.

This has suggested use of higher concentrated nitric acid (1:1, 1:2, 1:5) due to very slow leaching of cores and more diluted nitric acid (1:50, 1:100, 1:500, and 1:1000) to see the difference in leaching rate.

The core composition [2] will give out longer slurry working life and higher strength due to changes in magnesium oxide and fused silica mesh size. The higher concentration of nitric acid to water should also give quicker leaching rate than the more diluted nitric acid concentration.

5.2 CORE COMPOSITION [2] DISCUSSION

Core mixing: As shown in Figure 32, the addition of different percentages of magnesium oxide in Trial 4A suggests that magnesium oxide controls the setting of the mixed slurry. The magnesium oxide reacts very quickly with water to create magnesium hydroxide ($\text{Mg}(\text{OH})_2$). The magnesium hydroxide is a weak alkali which reacts rapidly with SiO_2 and plaster (calcium sulphate). This demonstrates that a greater percentage of MgO will result in a faster reaction and speedier setting.

5.2.1 CORE COMPOSITION [2] PROPERTIES AND REMOVAL

Trial A: Cores had moderate mean slurry working life, to allow for pouring into the wooden core box. Trial A was standardised to twelve seconds of slurry working life. In core composition [1], the Trial A had an average of four seconds of mean slurry working life. The mean slurry working life may not be adequate (depending on volume percentage) when related to slurry working life in commercial industries.

Trials 2A and 3A exhibited twenty-minute mean setting time, whereas Trails 4A and 1A had twenty-five and fifteen respectively. This shows that the plaster ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) works as the bonding agent for the core trials. Compared with the core composition [1], mean setting times in the wooden core box are not similar. This suggests that the slurry mix has a respective setting time according to the change in plaster and magnesium oxide in composition.

When removed from the wooden core box, the product from Trial 1A was weak and brittle compared with that from Trial 4A. The products from Trials 2A and 3A were hard, while that from Trial 4A was strong, with a smooth surface having no pores or

voids. All cores had splits and displayed uneven distribution of fused silica on their surfaces.

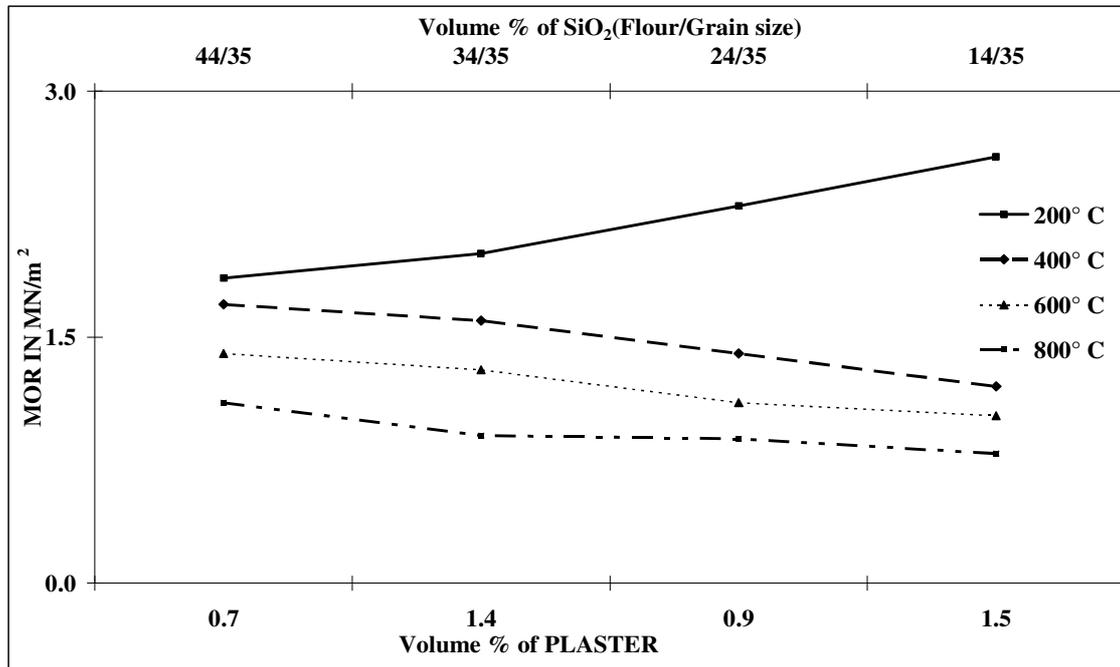


Figure 40. Mean MOR in Trial A at different fired temperature

Figure 40 shows the mean MORs of Trial A at different temperatures. At 200 °C, the product from Trial 4A has a higher mean MOR than those from other trials due the higher percentage of plaster and gave more stable forms. The plaster loses its water crystallization at just below 200 °C and anhydrous calcium sulphate is formed. At higher temperatures, plaster will become calcium sulphate, losing its bonding with the other composition materials. This results in lower mean MOR when fired at higher temperatures with different percentages of plaster.

The leaching of Trial A cores (at all fired temperatures) in diluted nitric acid (1:1, 1:2, 1:5) is greater than with other ratios (1:50, 1:100, 1:500 and 1:1000). It was observed that the higher the concentration of nitric acid to water, the faster the cores were leached. At four hours elapsed time, the cores inside the cast part were soft. Jet

washing would still be required to remove the residue. Visual outer surface examination noted that higher concentration of nitric acid had not had any effect on the cast part (Al- LM25) at any stage of the core removal process.

Trial B: The product from Trial 1B had similar slurry working life as that from Trial 4B, even though binder concentration was varied. This is due to the standard addition of magnesium oxide. There was no effect on the pouring of either of the core trials. Trial 4B slurry was high viscous than that in Trial 1B since water crystallisation was higher in latter. The mean setting time was quicker with higher percentage of water crystallization in core trials. Trials 2B and 3B had a similar mean setting time to the core composition [1]. Trials 1B and 4B had fifteen and twenty-five minutes mean setting time respectively.

The product from Trial 1B was weak and very “green” when removed from wooden core box. Extra care is recommended when manually handling 1B cores. The surface was rough and contained moisture. In Trial 2B the surface was harder than that in Trial 3B; that from Trial 4B was strong, having a smooth surface without any voids on the top. The uneven distribution of fused silica (30/50 setting on one side) was visible.

Figure 34 shows the mean MORs resulting from Trial B. Trial 1B (at all fired temperatures) recorded lower mean MOR than other core trials, due to higher water crystallization, which evaporated. The 4B resulted in higher mean MOR when fired at higher temperatures because of the binder (colloidal silica) which has greater resistance in higher temperature firing process. The 3B resulted in a higher mean

MOR than did Trial 2B at any temperature because of the higher percentage of binder. Cores produced using a higher percentage of binder offer a good benefit in investment casting, where ceramic cores must withstand the high melting temperatures of metals and super-alloys.

Dilute nitric acid (1:1, 1:2, 1:5) readily attacked the bonds in Trial B cores. Within four hours all of the Trial B cores were soft and turned into slurry. At all fired temperatures, higher concentrations of nitric acid have an extreme effect on the core. The other more diluted nitric acid (1:50, 1:100, and 1:500) took effect only after four hours. Products from Trials 1B and 2B, fired at 200 °C were soft by four hours, disintegrating in twenty-four hours. At other fired temperatures, cores were soft only after twenty-four hours. The diluted nitric acid at a ratio of (1:1000) did not have much effect on any core trial except in Trial 1B; this was soft after twenty-four hours. Trial B product has good leaching properties at different concentrations of binder which gives greater strength and rapid leaching in diluted nitric acid, a requirement for commercial applications. Trial B cores are highly recommended for use in commercial industry as they offer greater strength and quick-leaching properties.

Trial C: Trial 1C had no MgO, which gave longer mean slurry working life. When mixed, it had a very low viscous flow. The product from Trial 1C took forty minutes to set. The core was very weak and extra time was needed to dry. The mean slurry working life for Trial 3C was six seconds, while that for Trial 2C was ten seconds. Trial 3C showed quicker setting than Trial 2C, since it contained a higher percentage of magnesium oxide. Trial 3C cores took less time to set, small pores were visible on the outer surface when they were removed from the wooden core box. An uneven

distribution of fused silica (30/50) on the surface of cores was noted. Product from Trial 2C had no pores; the surface was rough and they were easy to remove from the wooden core box.

After firing at different temperatures, all cores of Trial 3C had cracks on their surfaces. This was due to the outer surface being quite dense. When fired at high temperatures the outer skin expands more than the inner material. These stresses cause cracks to appear on the surface.

Figure 35 shows the mean MOR of Trial C. Trial 2C also shows similar mean MOR to those of Trial 2A which has a similar compound formation. Difficulty in making Trial 3C resulted in lower mean MOR at different fired temperatures; the mixed core trial had set before pouring into the wooden core box was carried out. The mean MOR in Trials 1C and 2C is very similar to that of Trial 2A since all core compositions are same other than MgO percentage. In commercial industry, the slurry working life should be sufficient so that mixed slurry pouring can be carried out comfortably. Trial C cores have a limitation in slurry working life and setting time, which could have an effect in commercial applications.

Higher concentrations of nitric acid to water (1:1, 1:2, 1:5) attacked all the core trials within one hour. The cores were soft within four hours. The lower concentrations of nitric acid to water (1:50, 1:100, 1:500) had limited effect on Trial C. Only cores fired at 200 °C were affected after twenty-four hours. The lower concentrations of nitric acid to water (1:1000) had no effect on cores. Trial C cores have average strength and leaching at higher concentrations of nitric acid, which is not environmentally friendly.

Trial D: Trial 1D gave similar results as Trials 2A and 2C. Trial 2D had the standard slurry working life and setting time of twenty minutes. The surface was rough, had no pores and was easily removed from the wooden core box. The addition of higher percentage of binder gave a longer slurry working life, the addition of calcium silicate gave a longer core (dimensionally). Visually the dimensions (length) were longer than normal cores produced on other core trials.

As shown in Figure 36, the mean values of MOR of products from Trial 2D are higher than those of Trial 1D. Trial 2D had calcium silicate (CaSiO_3) bonding with plaster (CaSO_4) which makes it more robust. The elevated fired temperatures give higher mean MOR in Trial 2D, since the addition of higher percentage of binder and calcium to plaster generates di-calcium silicate. The di-calcium silicate, bonding with fused silica, will give tri-calcium silicate, which results in stronger compounds. Trial 1D product exhibited similar strength to that of Trial 2A. In relation to commercial applications, Trial 2D has a dimensional limitation, which may raise concerns about the accuracy of cast dimensions and shrinkage when fired.

Dilute nitric acid (1:1, 1:2, 1:5) has a greater effect on the Trial D cores at all fired temperatures. The other lower concentrations, (1:50, 1:100, and 1:500), did not have much effect on all core trials fired at different temperatures. After twenty-four hours, cores were soft; manual removal or jet wash is required to clean from the cast part. The higher concentration (1:1000) had no effect on core.

Trial E: Trial 1E involved use of fine casting plaster (FCP) which is a beta plaster similar to the dental plaster used in other core trials. In Trial 2E there was high viscous flow, because of there being denser particles of plaster. The setting time was twenty-five minutes for all Trial E cores. The 2E cores were dry, with smooth surfaces having no pores or voids. The uneven distribution of fused silica was observed when they were removed from the wooden core box.

Figure 37 shows the mean MOR of Trial E products fired at different temperatures. Trial 2E products have higher mean MOR than Trial 1E products, since the Crystacal R (CRL) alpha plaster has a higher density. The alpha plaster is made using higher percentage of water which makes the plaster denser and heavier. The Trial 2E product is denser, resulting in greater strength (mean MOR). At different fired temperatures, Trials 1E and 2E products have reduced strength due to the plaster losing its water content; the resultant plaster converting to dead burnt when fired at elevated temperatures.

As in other trials, Trial E cores showed the effect of the higher concentration of nitric acid to water (1:1, 1:2, and 1:5) at all fired temperatures. Within four hours, the cores were soft. The other diluted nitric acids (1:50, 1:100 and 1:500) had no effect in four hours but were soft within twenty-four hours at any fired temperature.

5.2.2 MEAN MOR COMPARISON

All core trials increased the mean MORs of products compared with core composition [1]. The fine mesh size of the fused silica (-200) has better particle packing than fused silica (-120) grain size. The surface bonding of finer particles is higher and bondings

are strong. Compared with the core composition [1], Trial A and D cores have more than 100% increase in mean MOR, Trial B and E cores have less than 50% increase in their mean MOR when fired at elevated temperatures. Trial C cores have a variation between 20 to 100% increase in mean MOR at different fired temperatures. The higher mean MORs on different core trials is useful in commercial applications during the waxing process on the ceramic core. Cores made by the moulding processes using a finer particle size produced better compaction of the particles, which increased the mechanical strength of resulting cores ⁽⁶⁰⁾. Since these cores are poured cores, the overall mean strength is less than the pre-formed cores, which are made by injecting the mixed slurry into core boxes under pressure.

5.2.3 CASTING WITH CORE COMPOSITION [2]

All core trials fired at different temperature were subjected to gravity die-casting. The single casting was done using mould with Al-LM25. The Al-LM25 used for core composition[1] was re-melted to use in core composition[2]. The re-melting was adopted to find the effect on aluminium with gravity die-casting. The aluminium melting temperature was attained to 680 °C.



Figure 41. Al-LM25 break since not feeding the metal properly

Figure 41 shows the Al-LM25 casting broken due to not feeding the molten metal properly. This is a casting defect, where not enough molten aluminium is present in solidification process. It is suggested that enough molten metal should be fed through the mould cavity. Extra care should be taken to make sure that metal is fully solidified.

The re-melting of the aluminium has no effect when re-cast using core composition [2] ceramic cores. The re-melting of aluminium alloy sand castings probably represents one of the worst cases since the oxide skin on a sand casting is particularly thick, having cooled from the high temperature in a reactive environment. The re-melting of aluminium alloy gravity die-castings⁽⁵⁴⁾ have an oxide skin that is much thinner and seems to give less problems.

5.2.4 REVIEW OF CORE COMPOSITION [2]

This section analyses the effects on ceramic core production when there is a change in fused silica particle size and a reduced percentage of magnesium oxide. The results suggest that finer particle sizes pack stronger than larger grain size fused silica. The finer particles will also produce smooth surfaces on the ceramic core and internal cast part. The use of grain size fused silica (30/50) should be discontinued, since not mixing properly and it settles in the bottom of the core. The lower percentage of magnesium oxide means that its reaction with silicon takes longer, enabling extended slurry working life.

The cores fired at higher temperatures do not leach with diluted nitric acid. The use of nitric acid (with higher concentrations) is very hazardous in commercial industry

and very difficult to dispose of. The lower concentrations of nitric acid may be implemented in commercial industries but a vacuum cupboard is needed (at extra cost) and separation of water/nitric acid should be performed before environmentally acceptable disposal is carried out. This has raised the possibility of a review into using an alternative leaching agent that is readily usable and environmentally friendly. The section has also raised other reviews of core trials in order to achieve a proper ceramic core.

The changes described in the next section will be to make cores with the finer particle size of fused silica, keeping the magnesium oxide constant at 0.1%, and firing at lower temperatures (of less than 400 °C). The core composition [3] should give a core with higher strength, longer slurry working life and leaching property. The batch sizes from ten to twenty-five were suggested to get more consistent strength of ceramic core for all core trials. Since Trials 2D and 2C have similar cores as Trial 2A, they are not considered as a part of further review. As an alternative to nitric acid, the use of diluted citric acid, acetic acid and potassium hydroxides as leaching agents are recommended since citric and acetic are environmentally friendly. The potassium hydroxide is used to leach out the ceramic cores from super alloys. The potassium hydroxide may attack aluminium but the experiment was carried out to find the leaching effect.

The core composition [3] will have flour size fused silica. The core trials will be fired at very low temperatures since higher temperatures do not have much effect on diluted acids. The alternative diluted acids should result in higher leaching property.

5.3 CORE COMPOSITION [3] DISCUSSION

Core Setting: As shown in Figure 42, the mean setting time is not altered by a change in core material particles. All core trials had longer slurry working life other than that in Trial C. In any commercial industry, the setting time should be less since ceramic cores need careful consideration regarding expansion and strength. The mean setting time of core trials were ideal for any commercial ⁽⁷⁰⁾ application, with the exception of that in Trial C.

5.3.1. CORE COMPOSITION [3] PROPERTIES AND REMOVAL

TRIAL A: The standard slurry working life for Trial A was fifteen seconds. The mixing process with binder and water was smooth. Visual observation showed that Trial 1A material had low viscosity, while Trial 4A material had very high viscosity due to its higher percentage of plaster. Trials 2A and 3A gave similar flow properties. This shows that higher percentage of plaster gives very high viscous flow to mixed slurry; this needs careful consideration when pouring into the wooden core box.

When removed from the wooden core box, Trial 1A cores exhibited a very smooth surface, indicative of too much moisture. The core was very “green” and manual handling was difficult. The Trials 2A and 3A cores were strong, while Trial 4A core was robust, with a dry surface. The manual handling of Trial A cores (with higher plaster addition) was easy. It was observed that distribution of the fused silica/plaster was even, with no voids or pores on the surface.

Figure 43 shows the mean MORs of Trial A cores; when cores were fired at 400 °C the mean MORs are very low compared with product at 200 and 300 °C. Trial 4A core at

200 °C had MOR of 4.11MN/m² due to the composition having a higher percentage of plaster. Other Trial A cores have reduced mean MORs when fired at more than 200 °C. In general, when plaster (CaSO₄·½H₂O) is heated to more than 200 °C, the strength reduces due to loss of water crystallisation. The MOR variations of cores when fired at different temperatures are expected, since adding different materials to dental plaster and water disturbs the strength and balance of its composition⁽⁶²⁾. Trial A suggests that plaster works as the bonding agent, giving strength to the core, with lithium carbonate possibly supporting the plaster after firing at moderate temperatures.

TRIAL B: Slurry working life was standardised for all the trials, no quick setting being apparent with higher percentages of water or concentration of binder. This suggests that reduced percentage of magnesium oxide has reduced quick setting in core trials. Trial 1B material had lower viscosity than Trial 4B material. The mean setting times for Trials 1B and 2B were both twenty minutes, even though the volume percentage of water was different. Trials 3B and 4B showed 2 minutes difference between them in mean setting times. This shows that binder (Ludox AM), in taking more time to react with water, creates a stronger bond with plaster.

After removal from the wooden core box, Trial 1B and 2B cores were weak, with a rough surface. Some small pores seen on the surface of Trial 1B product may be due to air trapped inside the wooden core box. Product from Trial 3B was strong, while that from Trial 4B was robust and had a smooth surface without pores or voids on the surface.

Figure 44 shows the results of Trials B, 1B and 2B showing lower mean MORs when fired at elevated temperatures with higher mean MORs on Trials 3B and 4B due to the change in water/binder concentration. As discussed previously, binders have higher resistance when fired to elevated temperatures. The binder and calcium in plaster made the cores stronger in Trials 3B and 4B when fired at 400 °C. Trial 4B recorded 5.04 MN/m² as mean MOR when fired at 400 °C.

TRIAL C: Trial 1C slurry had low viscosity, giving longer slurry working time than any other trials in core composition [3]. This was due to silicon dioxide taking longer to react with calcium sulphate to create a bond. Trial 1C had no MgO, while Trial 3C had 0.6% MgO. The setting times for Trials 1C and 3C were forty and fifteen minutes respectively. Because of quick setting on Trial 3C, pouring into the wooden core box was difficult, as was the production of cores. Trial C suggests that the lower percentage of MgO will give a longer setting time to mixed slurry.

Figure 45 shows the mean MORs of Trial C cores. The Trial 1C and 3C cores have higher mean MORs when fired at 200 °C. The Trial 1C and 3C cores have lower mean MORs when fired at more than 200 °C. This is similar to Trial A core mean strength (MOR). The addition of MgO does not have any effect on the MORs since added magnesium reacts with silicon. In commercial industry, to make cores while retaining control of slurry setting, strength and leaching is high priority. Trial C has suggested that an inconsistency in control of slurry working life and mean strength are not affected by the addition of other materials.

TRIAL D: The mixed slurry of Trial 2D was smooth and of high viscosity. Trial 2D gave a longer slurry working life after removal of air bubbles; its setting time was twenty minutes in the wooden core box.

After removal from the wooden core box, the core was hard with rough surface. The core was longer (length dimension) than in any other trials since calcium silicate was added. The cores were easy to handle manually, with no pores or voids on their surfaces.

Figure 46 shows the Trial 2D core mean MORs when fired at different temperatures. Trial 2D cores have higher mean MORs when fired at 400 °C. This is due to the higher percentage of Ludox AM and the addition of more calcium. Trial 2D suggests that higher percentage of calcium and binder (Ludox AM) will strengthen the core more than the addition of any other materials.

TRIAL E: The mixed slurry of Trial E had high viscous flow. Trial 2E slurry had higher viscosity than Trial 1E mixed slurry. The core setting times in the wooden core box for Trial E were thirty minutes each. The Trials 1E and 2E cores were hard; manual handling was easy. The surfaces were smooth and without any uneven distribution of fused silica on the surface.

Figure 47 shows that the mean MORs of Trials 1E and 2E cores are lower when fired at 400 °C due to plaster losing its bonding compounds and evaporation of water crystallization. Trial 2E cores have higher mean MOR than Trial 1E cores, due to high

density of the alpha plaster. Trial E cores demonstrate that addition of dense plaster will give a higher mean MOR when cores are fired at 200 °C.

Previous research work showed that the water-soluble ceramic cores were made with alumina (-200mesh size) and zircon (-120mesh size). The cores were fired at 200 , 400 , 600 and 800 °C for two hours. Sodium hydrogen phosphate⁽⁸¹⁾ was used as binder. The ceramic cores strength was recorded from 5.6 to 0.69 MN/m² as final MORs fired from 200 to 800 °C. These water-soluble ceramic cores were used in gravity die-casting and investment casting of aluminium alloys.

The Trial 4B cores which were produced using binder (Ludox AM) fired at 800 °C have MOR of 5.04 MN/m² with fused silica (-200 and -120 mesh). This suggests that plaster and binder (Ludox AM) produce higher strength when fired at 400 °C.

CORE REMOVAL

POTASSIUM HYDROXIDE REACTION

Figure 50 shows the reaction of diluted potassium hydroxide with Aluminium LM25. Potassium hydroxide dissolves readily in water with the vigorous generation of heat. The reaction of potassium hydroxide with aluminium forms potassium salts, in which the aluminium is incorporated into a negatively charged ion; the core aluminium turns black in colour. Since the dilution with water is much less, the time taken to affect the aluminium was longer. The cores were readily attacked by the diluted potassium hydroxide within four hours. The leaching process was discontinued since as anticipated, the aluminium surface was attacked.

Acetic Acid (1:1): The higher concentrations of *acetic* acid in water (1:1) attacked cores. All cores (fired at different temperatures) are affected within one hour making cores soft. After four hours the cores were very soft and readily attacked by the diluted acid, showing that acetic acid reacts with the sulphur (components of plaster), attacking the bonds and weakening the core.

Acetic Acid (1:100): The less concentrated acetic acid (1:100) took one hour to attack the bonds for cores fired at 200 and 300 °C. At 400 °C, the cores were soft in four hours, suggesting that dilute acetic acid took time to break bonds by attacking sulphur. This solubility, in conjunction with its miscibility, makes acetic acid a widely used industrial chemical. Even though acetic acid is often considered a relatively weak acid, diluted acetic acid can dissolve and soften ceramic compounds such as salts and other elements.

Citric Acid (1:1): With higher concentrations of citric acid to water ratios, all cores were soft within one hour. The cores were very soft and brittle; the higher concentration of citric acid reacts with calcium, producing mild heat that helps to break the bonds with other compounds. The “exothermic process” reaction has no effect on aluminium since the oxide layer on aluminium surface protects from any reaction with acid and water.

Citric Acid (1:100): The cores fired at 200 °C were attacked in four hours. Citric acid took a longer time to attack the calcium compounds. The cores were soft, but needed a manual removal process. After twenty-four hours, the cores were weak and brittle, suggesting the reaction with diluted acid is prolonged. The use of citric acid to remove

ceramic cores in commercial industries offers advantages in both environmental and financial aspects.

5.3.2. SEM IMAGING

Trial 4A core surfaces fired at 200 °C were examined using scanning electron microscopy (SEM). The images of Trial 4A core surface with different fused silica mesh size used in core composition [3] and [1] are shown in Figures 48 and 49. The fused silica (-120/30-50) has a rough surface, influencing internal finish of the cast part, by inhibiting the flow of molten aluminium. This will create very high stress concentration points on the surface leading to cracks and pores. These pictures suggest that Fused silica (-120/30-50) used in core composition [1] will produce rough and uneven surfaces on cores. These peaks on core surfaces were seen on different core SEM images that were fired at different temperatures.

5.3.3 CASTING WITH CORE COMPOSITION [3] CORES

All cores trials were cast with Al LM25 metal. There were no defects found on the outer surface of the cast part. Since the molten metal has direct contact on to the core, gases are released. Since no airing holes were designed in the mould, gases released from the core were trapped inside. These gases can have a catastrophic effect on aluminium creating hot tears and other surface defects.

In commercial industry, putting a coil of copper tube (perforated in several locations along the length) inside the core investment within the cast cavity is used to vent the gases. This will allow liberated gases to be expelled from the core itself when the metal

flows in around the core. Not venting the core can cause the appearance of melt not completely filling the cavity.

5.3.4 REVIEW OF CORE COMPOSITION [3]

The core composition [3] results and discussions have shown that the use of finer mesh sizes (-200/-120) of fused silica provides better packing with other core materials, which also increases the strength of cores with smooth surfaces. The core composition [3] demonstrates that mixed slurries have a standard slurry working life. As discussed, the addition of magnesium oxide controls the setting of mixed slurry. The core composition [3] has shown that adding 0.1% of magnesium oxide gives moderate slurry working life.

The mean MORs of the core from trials increased by more than 20% due to changes in fused silica particle size. The use of finer particles of fused silica improved the mix and produced a smooth flowing, easy pouring slurry, with acceptable setting and removal from the wooden core box.

Since the use of nitric acid is not environmentally friendly, citric and acetic acid were adopted to leach out the cores. Higher concentrations of citric and acetic acids attacked and leached the core trials in less time. Lower concentrations of acid took a longer time to leach the cores.

Results from core composition [3] lead to the conclusion that a reasonable and feasible ceramic core can be made with relevance to the specific requirement of commercial industry. Since smooth surface and higher strength resulted from the use of fused silica

finer mesh size, further core compositions were formulated without lithium carbonate, in order to see the effect on bonding and strength. Even though the setting time without MgO is forty minutes, cores were produced. This has influenced the recommendation to remove MgO from all core trials to investigate the impact on core mix and setting. The leaching of core trials is higher in Trial 1B when fired at different temperatures, reference to Tables 31 to 33.

The ceramic core was made using core composition [3] without any major concerns about mixing, core strength or leaching from the cast part. The core composition [4] will be fired to more than 400 °C in the new core trials, to examine the leaching ability from diluted citric and acetic acids. The removal of magnesium oxide and lithium carbonate will remove the Trial C and 1D cores since they are similar to results in Trial 2A. The aim of the core composition [4] will be to consider the dimensional accuracy and shrinkage of core trials at different fired temperatures.

5.4 CORE COMPOSITION [4] DISCUSSION

The composition [4] had an average of thirty seconds of dry mix and thirty-five seconds of mean slurry working life after mixing (binder/water mix). This suggests a homogenous mix on all composition [4] mixes. The usual pouring process was adopted without any limitations on filling the wooden core box. Trials 3B, 4B and 2D gave a higher viscosity than other core trials since the binder concentrations were higher. Adding a high percentage of binder to the ceramic raw materials⁽⁶³⁾ makes the slurry flow very smooth and slow. Since the water to binder ratio was equal in other trials, these core trials had lower viscosity than the rest. In industry, the viscosity of mixed slurry depends on the shape and application of the ceramic core. For long and thin ceramic cores such as those used for turbine blades, a lower viscosity would be expected. The lower viscous flow of mixed ceramic materials is preferred when dimensional sizes are large.

Figure 52 shows the mean setting time of the core trial in the wooden core box. The core trials have longer setting times since no magnesium oxide was added. The reaction between magnesium oxide and water was not present, resulting in a longer mean setting time. The reaction between the binder/water and calcium in plaster achieved the setting time of the core trial.

After removal from the wooden core box, a smooth surface on the core was visible. This is due to use of finer fused silica particle. The core Trials 1A, 2A, 3A, 2B, 3B, 4B, 2D, 1E and 2E had forty minutes of setting time; Trials 4A, 1B, 4B and 2D had thirty-five minutes. This suggests that a higher percentage of calcium in core trials has an effect on the mean setting time. In addition, the binder with magnesium oxide has a

very significant advantage in quickly setting in the wooden core box. The ceramic castings produced for dental prostheses ⁽⁶⁴⁾ have only four to ten minutes of setting time. The core composition has 50% to 80% of magnesium oxide, 10% to 40 % of alumina and small percentages of other ingredients mixed with Ludox (Colloidal silica). The mix sets very quickly, is fired for two hours.

5.4.1 CORE COMPOSITION [4] MEAN DIMENSIONAL CHANGES

In general, it was found that the core composition [4] cores showed greater contraction in both length and diameter when fired at higher temperatures. The mass transmission (loss of weight) is higher in the direction of diameter than length. The evaporation of water crystallization suggests material is pulled together to centre of the core. The evaporation comes off in one direction, through the diameter. The core trials have not exhibited any thermal shock since fused silica and plaster have a very low thermal expansion and the cores were dried at room temperature before firing to their respective temperatures and cooling for two hours from respective fired temperatures.

In commercial industry, two firing processes are carried out to prevent thermal shock on ceramics. The first process is at low temperature (~120 °C), to remove the excess moisture and binder material. This low temperature firing results in less dimensional change during the final process. The higher temperature process (above 871 °C) is performed to sinter the material, hardening the ceramic particles; this ensures the removal of any moisture in the ceramic cores.

One firing cycle is also used in casting industry; ceramic cores are produced overnight, dried at a constant temperature (mostly room temperature), then fired to elevated temperatures. The ceramic cores that have one firing cycle⁽⁶⁶⁾ are mostly fired above 1300 °C. The higher temperature confirms the smaller dimensional change and hardening of the ceramic material.

The core composition [4] cores had a single firing cycle after twenty-four hours drying at room temperature. The addition of plaster and use of one firing cycle at different temperatures has resulted in contraction of both length and diameter. The core trials have a greater accuracy with regard to dimensional tolerance. Depending on the end application and the tolerance of dimensional contraction, the core composition [4] cores can provide excellent ceramic cores.

Figure 53 shows that Trial A has less length contraction percentagewise with higher percentages of plaster when fired at 200 °C. Trial 4A has 0.02% contraction, while 1A has 0.10% contraction at 200 °C. When fired at higher temperatures, Trial A has a greater percentage of length contraction. At 400 °C and 600 °C, Trials 3A and 4A cores gave rise to 0.2% length contraction.

Figure 54 shows that Trials 1B and 4B had less contraction. This is due to the fact that in Trial 1B water crystallisation evaporation occurred, while in Trial 4B there was higher thermal resistance from the binder. At 800 °C, Trial 3B has 0.22% length contraction which is greater than in Trial 4B. This may be due to it being less dense, resulting in non-uniform dimensional contraction. Trial 2B showed linear thermal contraction, showing greater contraction when fired at elevated temperatures.

Figure 55 shows that Trial 2D has less length contraction compared with any other trials when fired at different temperatures. The higher percentage binder, which has thermal resistance (Ludox AM) when fired at higher temperatures, has reduced the percentage of length contraction.

Figure 56 shows that Trial 2E has a higher percentage of length contraction than Trial 1E. The alpha plaster (CRP) contracts more (one firing cycle) when fired at any temperature than beta plaster (FCP). At any fired temperature, an average rise of 0.03% in length contraction was recorded in Trial 2E. Trial 1E was produced with fine casting plaster, which is a beta plaster similar to dental material.

Figure 57 shows that the shrinkage percentage in Trial A core diameters are high when fired at elevated temperatures. The Trial 4A has much less diametric shrinkage than Trial 1A. The 2A and 3A trials have an average of 0.8% increase in diametric shrinkage when fired at different temperatures.

Figure 58 shows the diametric shrinkage percentage of Trial B. Trial 4B has 0.24% contraction compared with 0.12% of Trial 1B at 200 °C. At other fired temperatures, Trials 1B and 4B show linear diametric shrinkage. At 800 °C, Trial 3B exhibits 0.69% of diametric shrinkage higher than Trial 4B. This suggests similar analysis to that of Trial B length contraction, where non-uniform dimensional contraction occurs.

Figure 59 shows that Trial 2D has high diametric shrinkage when fired at different temperatures. At 800 °C, Trial 2D has 0.69% of shrinkage compared with 0.44% at

200 °C. It is also observed that an average of 0.8% increase in diametric shrinkage occurs when fired between 400 to 800 °C.

Figure 60 shows the Trial E diametric shrinkage, which was much higher than any other core trials. The Trial 2E has 0.8% of diametric shrinkage at 800 °C. The shrinkage is due to having one firing cycle when fired at higher temperature.

In the investment casting process, the ceramic core is required to maintain close dimensional tolerances. Depending on the particular core geometry and the intended application, it may be approximately 0.01-0.02 mm. Conventional silica/binder-treated cores⁽⁶⁶⁾ exhibit a high degree of core distortion (e.g., 0.2-0.5 mm), which frequently exceeds the dimensional tolerances. The highest length contraction percentage was on Trial 2E at 800 °C with 0.32% since alpha plaster contracts high; this relates to a change in length of 0.48 mm difference from original to final length.

The addition of several other materials to the fused silica (93% pure) has resulted in very small dimensional changes⁽²²⁾ with low and sometimes constant linear thermal expansion between 500 and 1100 °C. The higher coefficients of thermal expansion resulted when fired between 1350 to 1525 °C. The ceramic cores were also cooled at 900 °C under stress conditions. The results indicate that there is very little effect on the ceramic cores dimensional expansion or contraction. The fused silica crystallisation effects occur during heating to the casting temperatures⁽²²⁾ of super alloys.

The ceramic cores produced from fused silica have a small influence on expansion or contraction behaviour. Researchers elsewhere examined six different silica cores ⁽⁶⁵⁾ fired at 1530 - 1600 °C and held at maximum temperature for ten to thirty minutes prior to cooling to room temperature. The experiments show that heating or cooling of the silica core has caused much less shrinkage when fired at higher temperature than lower temperatures. The cores had two firing cycles.

Normally, ceramics have a low linear co-efficient of thermal expansion. The strong *interatomic* bonding forces ⁽⁶⁷⁾ found in ceramic materials give a value for the coefficient of thermal expansion in a range between $0.5-15 \times 10^{-6}$ (per °C). An interatomic expansion produces relatively small macroscopic dimensional changes, explained by a low atomic packing density.

With regard to temperature dependence, the linear coefficient of expansion rises ⁽⁶⁷⁾ with an increase in firing temperature. The fused silica has a value of 0.4×10^{-6} (per °C) at room temperature. The beta (dental) and alpha (Crystal R) plasters resulted in expansion of 0.3% and 0.5% after two hours setting in room temperature.

5.4.2 CORE COMPOSITION [4] PROPERTIES AND REMOVAL

The core composition [4] ceramic cores have provided sufficient strength (MOR) in relation to compositional additions and shape produced. The composition [4] has demonstrated that use of accelerator is not required to improve the strength of the core.

Trial A: Figure 61 shows that the Trial A cores have higher mean MORs at 200 °C but their strength is reduced when fired at elevated temperatures. The Trial 4A recorded a value of 4.9 MN/m² at 200 °C, which was higher than any other trial A cores. This suggested that the presence of calcium improves the effectiveness of the binder when fired at lower temperatures.

Trial B: Figure 62 shows the Trial B cores having lower mean MORs in Trials 1B, 2B and higher mean MORs resulted in Trials 3B and 4B when fired at higher temperatures. The higher binder concentration in core trials has shown that stronger bonds are active with silica. Trial 4B recorded a value of 4.98 MN/m² at 800 °C. This is due to the binder having thermal resistance when fired to higher temperatures. Even at 200 °C, Trials 3B and 4B have higher mean MORs when compared with Trials 1B and 2B. A minimum 30% increase in MORs is seen in Trials 3B and 4B cores when fired at elevated temperatures.

Trial D: Figure 63 shows that Trial 2D had higher mean MORs when fired at elevated temperatures. The core had additional calcium silicate (CaSiO₃) which made the core robust and strong. The reaction between plaster and calcium silicate gave increased strength. At 800 °C, 5.2 MN/m² been recorded as mean MOR. At lower fired temperatures, Trial 2D recorded a higher mean MOR, similar to 4B.

Trial E: Figure 64 shows that Trial 1E core (beta plaster) has a lower mean MOR than Trial 2E core (alpha plaster). The mean MOR is higher in the alpha plaster, since it is highly dense and a quick observation uncovers water crystallisation. The Trial 2E exhibits a minimum of 5% increase in mean MOR when fired at different

temperatures compared with Trial 1E cores. The plaster data sheet ⁽²⁷⁾ shows that alpha plaster resulted in higher strength than the beta plaster after setting for two hours.

The core composition [4] cores are “performed core”(poured to wooden core box), where slurry is poured into the wooden core box. Due to reaction between materials, the mixed slurry sets in a relevant time. This will have a lower mean MOR than the pre-formed cores, where mixed slurry was injected into the wooden core box under pressure, resulting in higher packing between particles.

The preformed (injected to mould) ceramic core produced with 59% fused silica (particle size-116 μm) and 40% of binder (high density polyethylene wax) fired with two-firing cycles⁽⁶⁰⁾ records a MOR of 10.9 MN/m^2 . The dimensions of the core after the firing cycle were 511.8 mm in length and 49.7 mm in breadth and had a weight of 350 grams. Sintering was at 1200 °C for five hours followed by cooling to room temperature.

Ceramic cores produced by silicon nitride and aluminium nitride ⁽⁷⁰⁾ have recorded higher MORs when fired at higher temperatures. There were two different grain sizes used to produce the ceramic core slurry (50/315 and 400 μm particles) mixed with binders. A two firing cycle was exhibited, initially at 100 °C and finally sintering at 500 °C. The cores recorded MORs of more than 8.0 MN/m^2 , due to them being impregnated in an organically based resin after firing.

Trial 2D produced an MOR of 5.89 MN/m² with one firing cycle to 800 °C for two hours, followed by cooling to room temperature for two hours. The core also has binder (Ludox AM), which is normally amorphous silicon oxide. This demonstrates that the new materials have better MORs than those previously recorded with different refractory materials.

CORE REMOVAL

Table 34 shows the effect of diluted citric and acetic acid (1:1), reacting with all trial cores within one hour. The cores fired at lower temperatures showed much more effect (softer) than cores fired at higher temperatures. At all fired temperatures, Trials 1B and 2B were effected due to having a lower concentration of binder. The addition of less binder (Ludox AM), exposed the adhesive bonds of compounds in core trials. The reaction to the surface may take a longer time since fused silica may have some glassy surface after firing process.

The ceramic cores made with aluminium nitride were leached out with an 18% citric acid aqueous solution, taking an average of seventy minutes⁽⁷⁰⁾ to do so. The research also shows that adding calcium carbonate to the core material increased the leaching rate by more than 50% in different aqueous solutions. The alumina cores produced for investment casting also leach out with very slow rate in diluted potassium or sodium hydroxide. The cores were fired at higher than 1000 °C. It is claimed that the higher density of ceramic cores reduces the leaching rate in the aqueous solutions (diluted acids). The leaching rate is about 10 mm/h with 20-25% of density⁽⁷¹⁾.

The leaching rates with Trial 1B when fired at lower temperature are higher than those previously claimed. The cores were soft within one hour and broke within four hours. The leaching rate may be suitable for commercial applications, but with a higher concentration of citric or acetic acid the rate can be reduced.

5.4.3 CASTING WITH CORE COMPOSITION [4]

The gravity die-casting was carried out with core composition [4] ceramic cores. Aluminium was melted to a constant 680 °C. The contraction in aluminium is assumed to be about 3% to 7% once solidified after reaching melting point. It has noted that aluminium alloy castings of 300 mm or more internal diameter exhibit a patternmaker's contraction considerably less than that which would have been expected for an external system. This seems almost certainly to be the result of the expansion of the internal core as a result of the extra heating from the internal running. For a silica sand core, this expansion can be between 1.0 and 1.5 %, effectively neglecting the shrinkage allowance, which is normally between 1.0 and 1.3 %.

The composition [4] cores have less than 1% contraction. This suggests that final diameter after removal cast part will be the core diameter after firing. This suggests that cores have less shrinkage compared with acceptable tolerance on cast part shrinkage standards from industry.

5.4.4 REVIEW OF CORE COMPOSITION [4]

The core composition [4] ceramic cores were intended to produce a core without an accelerator or the addition of magnesium oxide. The core composition [4] has shown that magnesium oxide is needed to moderately set the reaction between binder/water and other materials. The magnesium oxide reaction will increase the slurry setting time. lithium carbonate can be removed from the next core composition since the cores have exhibited higher strength

The addition of less binder with a higher percentage of water leads to a faster leaching rate in core trials. It also has good strength, shrinkage and leaching rates when fired at different temperatures. The core composition [5] will consider the addition of magnesium oxide to the core trials. The binder/water concentration will also be changed for all core trials. A lower ratio of binder with higher ratio water will be added to all core trials to evaluate the effect on strength, dimensional change and leaching property. The application of binder/water concentration to all core trials will give cores similar to Trials B, C and 1D. These are removed from core composition [5]. The core trials are subjected to more mechanical properties. The core composition [5] will involve Weibull analysis and fracture toughness properties. This will provide the material resistance to fracture and survival probability relation to strength of core.

5.5 CORE COMPOSITION [5] DISCUSSION

The mixed slurry had low viscosity. The flow of slurry was fast, since water made the mix less dense. A high amount of air bubbles were formed when the mixing process was carried out. Before pouring, air bubbles were removed from mixed slurry to make sure that no pores occur on the external surface. The addition of higher concentration of water than binder (Ludox AM) has made the core trials set quickly in the wooden core box. Figure 65 shows the mean setting time of composition [5].

Trials 1A, 2A, and 1E had twenty-five minutes, Trials 3A, 4A, and 2E had twenty minutes of mean setting time. As previously discussed, the higher percentage of plaster reacting with water sets faster, than lower percentage of plaster in core trials. Since, Trial 2E material was made with high dense plaster (alpha), the reaction with water was quick. The Trial 2D material had twenty minutes as mean setting time. The addition of plaster with calcium silicate had an instant reaction with water to form the bonding.

The compositions used in core composition [5] had suitable mean setting times. The addition of water/binder ratio gives low viscous mixed slurry, which is an advantage in producing long and thin ceramic cores in commercial industries.

5.5.1 CORE COMPOSITION [5] MEAN DIMENSIONAL CHANGES

The composition [5] core mean diameter shrinkage or mean length contraction percentage varied about 10% compared with composition [4]. The changes of mean length and diameter are obvious due to the ceramic behaviour when fired at higher temperatures. The changes in mean diameter or length have no effect on the addition

of magnesium oxide. The mass transmission (loss of mass) is still through diameter rather than length. The materials are contracted to the centre of the core, where loss of mass has occurred. Again, ceramic cores have not exhibited any thermal shock, since cooling is done in the muffle furnace for two hours.

Figure 66 shows the length contraction of Trial A. The material with higher percentage of plaster contracted more at 800 °C. There is minimum 0.02% increase in length contraction from 600 to 800 °C on Trial A. Trial A suggests that when cores are fired at elevated temperatures, the length contraction percentage are reduced.

Figure 67 shows the Trial 2D length contraction percentage, 0.04% increase in length contraction from 600 to 800 °C. The 'green' cores were longer than any core produced in composition [5] trials due to the addition of calcium silicate.

Figure 68 shows the Trial E length contraction percentage; Trial 1E shows less contraction than Trial 2E. From 600 and 800 °C, the contraction of 0.08% recorded is higher. The Trial 2E cores are made using alpha plasters, which shrink more than beta plaster when fired at elevated temperatures.

Figure 69 shows the diameter shrinkage percentage in Trial A. At 800 °C, Trial 4A has 0.86% shrinkage compared with 0.59% in Trial 1A. From 600 to 800 °C, there is a 0.1% increase in diameter shrinkage on Trial A. This suggests that diameter shrinkage is much less when ceramic cores are fired at elevated temperatures.

Figure 70 shows the Trial 2D diameter shrinkage percentage, which has no difference in shrinkage. Both the cores fired at 600 and 800 °C gave 0.6% of diameter shrinkage. This suggests that after a two-hour firing cycle, the two bonding systems are stronger when fired at elevated temperature.

Figure 71 shows the Trial E diameter shrinkage, Trial 2E shows 0.8% increase in diameter shrinkage recorded between 600 and 800 °C. This is due to high dense plaster having higher expansion when fired at elevated temperatures.

The length contraction and diameter shrinkage of core trials are excellent compared with any other core materials used to make ceramic cores. It has much lower contraction than other ceramic core materials⁽⁷⁵⁾ used commercially.

Ceramic cores produced with silica material with an addition of 0.05% of MgO gave 2% shrinkage with synthetic amorphous silica (binder). The amount of core shrinkage varies directly with the percentage of synthetic amorphous⁽⁷⁵⁾ silica in the core material slurry. If the core is formed of a similar core material, slurry without synthetic amorphous silica will probably have approximately 0.2 percent shrinkage⁽⁷⁵⁾ during firing (two firing cycle).

Ceramic cores produced with silica (17%), zircon, (30%) and binder (colloidal silica gel) have 0.5% of shrinkage⁽⁷⁶⁾ in dimensions. This is from two firing cycles. The advantages of two firing cycles are a complete removal of water of crystallization in one cycle, and removal of binder in the final sintering process.

5.5.2 CORE COMPOSITION [5] CORE PROPERTIES AND REMOVAL

The core composition [5] cores recorded lower mean strength (MOR). This is due to a low addition of binder in making the core. The core composition [5] cores have demonstrated that use of less binder and high water can produce a core able to withstand a higher sintering process, of sufficient strength and leachable property.

TRIAL A: Figure 72 shows Trial A mean MORs when fired at different temperatures. The mean MOR increased with a higher percentage of plaster when fired at 600 °C. The Trial 1A cores have a low mean MOR of 1.1 MN/m² at 800 °C due to a much low percentage of plaster. The plaster works as the one-bonding agent in the core trial. The mean MORs increased with a minimum 0.2% with increase in plaster percentage in core trials.

Trial 2D: Figure 73 shows the Trial 2D core mean MORs when fired at different temperatures. This trial has a two-bonding agent. The core has additional calcium silicate to produce the bonding with plaster. At 600 °C, 2.2 MN/m² is the highest mean MOR recorded by any other core trial in core composition [5]. At 800 °C, Trial 2D core recorded a MOR of 1.93 MN/m². This suggests that addition of calcium compounds produces high strength in the core.

Trial E: Figure 74 shows the Trial E core mean MORs when fired at different temperatures. At 600 °C, Trial 2E core has a MOR of 1.70 MN/m² and Trial 1E core has a MOR of 1.35 MN/m². This is due to high dense plaster producing strong bonds. The mean MORs of Trial E cores are reduced by a minimum of 0.1% at 800 °C. This

is a small reduction in mean MOR when compared with commercial ceramic cores that are fired at elevated temperatures.

The core composition [5] cores suggest that adding a lower percentage of binder results in suitable mean MORs. The ceramic cores have sufficient strength for manual handling and withstand casting temperatures of non-ferrous metals. The cores also exhibited a low viscous flow and adequate slurry working life. In commercial applications, the desired mean MORs can be achieved with the addition of binder to core trials, depending on the application.

CORE REMOVAL

Table 38 shows that diluted citric and acetic acids (1:1) readily affected the core composition [5] cores. The low binder addition has diluted acid attacking bonds in less time. The cores begin to soften in one hour. The cores were very soft after four hours. Manual removal may be needed for all core trials. After twenty-four hours, cores were too soft due to salt formulation. The core collapsibility (removal property) is higher when a low amount of binder is added to core trial. This suggests that less binder in producing the core gives quick leaching rate but suitable strength, which can withstand the casting temperatures of non-ferrous alloys.

5.5.3 FRACTURE TOUGHNESS (K_{IC}) AND CRITICAL DEFECT SIZE

Due to the brittle nature of ceramics, it is assumed^(80, 91) that the manual notches or cracks, although relatively large at average “6 mm” are semicircular. If the crack or notch is having a cutting edge (sharp), the degree of stress concentration⁽⁸⁰⁾ is lower and may give spurious values.

The 'Y' parameter relates to the shape of the notch or crack, specimen geometry and test configuration. Most commonly 'Y' is the ratio of 'a/D' where 'a' is the crack length and 'D' is the diameter of solid bar. Because determining the notch shape (they are not exact part-ellipses) is impossible, 'Y' is only an approximate. Tables 35 and 36 shows 'K_{IC}' at different temperatures on core trials.

At 600 °C, Trial cores 1A and 2A recorded K_{IC} values of 0.11 MN m^{-3/2}. The K_{IC} for Trial 1E cores is similar to that in Trial 1A and 2A. Core Trials 3A and 2E gave a value of 0.14 and 0.13 MN m^{-3/2} respectively. Trials 4A and 2D recorded a value of 0.16 MN m^{-3/2} and 0.15 MN m^{-3/2} respectively; these are the highest K_{IC} values. At 800 °C, all core trials had very similar K_{IC} values as at 600 °C. Trial 1A gave a value of 0.08 MN m^{-3/2}. Trials 3A, 4A and 2D gave a value of 0.14 MN m^{-3/2} and Trials 1E and 2E gave 0.11 and 0.12 MN m^{-3/2} respectively.

The plain strain fracture toughness⁽⁷⁸⁾ of advance ceramics can be as low as 3 MN m^{-3/2} and as high as 12 MN m^{-3/2}. This is due to fracture strength being recorded in the range 350-1050 MN/m². The sintering temperature (firing) is above 1300 °C. The materials have higher strength with higher percentage of binder in producing them such as alumina, zircon.

The K_{IC} at room temperature of fused silica⁽⁶⁷⁾ is 0.79 MN m^{-3/2}. The core trials were fired at 600° and 800 °C, cooled for two hours at each temperature. The mean fracture MORs are also very low. The addition of different materials, chemical bonding and firing cycle may have affected the low K_{IC}. A high value of K_{IC} may be achieved with high value on 'Y' and MOR by using Equation 10.

CRITICAL DEFECT SIZE

Tables 37 and 38 show the critical defect size at different fired temperatures on core trials. The mean critical defect sizes on cores when fired at 600 and 800 °C are similar. The mean critical defect size has increased in Trials 3A, 2D and E when fired at 800 °C. Normally, critical defect size of ceramic materials should be less at higher temperatures.

The particle size value for fused silica -200mesh and -120mesh (74 μm and 125 μm) are 0.074 and 0.125 mm respectively. The particle size of plaster (BpB formula) is 0.040 mm (40 μm). For cores fired at 600 and 800 °C, the mean K_{IC} and fracture MORs from trial 2D has a critical flaw size of 0.55 and 0.58 mm. This is high compared with the particle size of fused silica and plaster. The particle size and critical defect size suggest that the shape of critical size is not possible to define. A further analysis is needed to prove the shape of critical defect size that will help in finding the crack initiation. The use of scanning electron microscopy and X-ray diffraction can help in providing the dimensions of critical defect size.

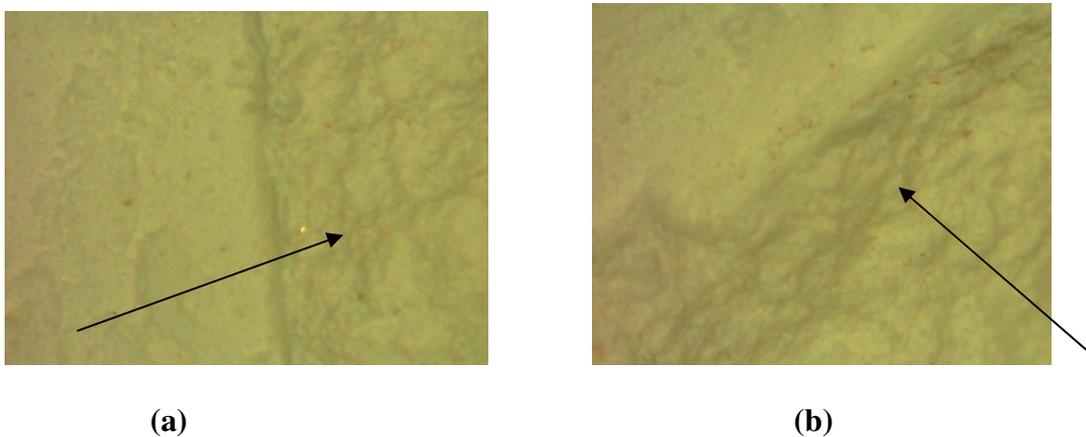


Figure 79. (a) Crack on notched core Trial 3A at 800 °C, (b) Crack on notch core Trial 3A at 600 °C.

Figure 79 (a) shows the crack after three-point bend test on notched core Trial 3A at 800 °C. The straight line is the depth of notch. As observed, the cracks have interlinked after the load applied by bending moment. The cores have micro cracks, which have propagated by transmission to notch depth. The path of the crack front will also follow the direction in which least energy is required to propagate it.

Figure 79(b) shows the crack after bending on notched core 3A fired at 600 °C. The observation suggests that cracks have not elongated. It has become smaller; packed cracks were observed in a hand-held microscope. The observation suggests that the crack process appears to have sub critical crack growth. The ceramic core is subjected to loads below its materials strength, and particularly if water or moist airs are present in its environment, a crack can start from a critical defect, growing very slowly at first, then accelerating under further load until failure occurs.

This observation was found on all different cores fired at 600 and 800 °C. This suggests that ceramics mixed and poured (with no injection or force used) to produce a circular bar have characteristics of sub critical crack propagation when fired between 600 and 800 °C. The observation suggests that semicircular notch bars subjected to bending load tend to grow asymptotically. The convergence is more rapid on crack depth surface.

5.5.4 WEIBULL ANALYSIS OF MOR VALUES

According to Section 2.5.3, The Weibull shape (β) is greater than 1.0 suggesting that probabilities of failure rate are higher. Table 40 shows the Weibull scale and shape for core trials at different firing temperatures. A Weibull Shape (β), having a value higher

than unity, would indicate that the material contained flaws. These parameters are not true material parameters but are dependent on test conditions, e.g. temperature of firing.

TRIAL	600 °C			800 °C		
	Shape(β)	Scale(α)	C orr	Shape(β)	Scale(α)	C orr
1A	1.3	0.3	0.98	1.3	0.1	0.98
2A	1.3	0.2	0.97	1.3	0.1	0.97
3A	2	0.5	0.99	2	0.3	0.99
4A	1.1	0.4	0.98	6.8	0.8	0.99
2D	4.5	0.7	0.99	1.5	0.3	0.98
1E	1.9	0.2	0.99	2.6	0.3	0.99
2E	2	0.3	0.99	1.3	0.3	0.95

Table 40. Weibull shape, scale and Correlation coefficient for cores fired at 600 and 800 °C

FigureS 75 to 78 show the distribution overview plot for cores fired at 600° and 800 °C. The Minitab® is used to calculate Weibull scale (β), shape (α) and correlation coefficient. The correlation coefficient (C orr) is a fractional value describing how well the regression equation fits the experimental data, a value close to unity indicates near perfect prediction.

The great variation in strength (MOR) of identical specimens requires testing large numbers of samples (>50) to achieve truly representative values. The Weibull analyses were limited to applications on cores fired at 600 and 800 °C temperature due to higher variable observed from other core trials when fired to less than 400 °C. A further study is required to find out the suitable application of Weibull in core failure analysis.

5.5.5 REVIEW OF CORE COMPOSITION [5]

The composition [5] has suggested that diluted citric and acetic acid have leaching effect on cores when fired at 600 and 800 °C. The addition of magnesium oxide has increased the mean setting time. This suggests that mixed slurry of core trials are controlled by magnesium oxide, water and binder. The high water addition results in lower mean MORs in different core trials when fired between 600 and 800 °C. The mean MORs on different core trials was sufficient to withstand casting temperature of aluminium.

The mechanical properties of core trials have suggested that core trials have better strength, very low fracture strength and low critical defect size. This suggests that cores have micro cracks and internal pores. The Weibull analysis has suggested that most of the core trials have very low survival probability with reliable values (C_{orr}).

The core composition [5] has suggested that water based cores can be produced which have good strength and leaching property for aluminium. The future review of the core trial will be on water/binder addition on core trials. The core composition [6] will be reviewing the cores produced with only binder in core trials and [7] with water. This observation will define the strength of core materials when fired at different temperatures. The core composition [5] make up will be used due to better understanding of filler and bonding material behaviour at different core trials. The water addition core should provide very low MORs compared with binder in core trials. The cores made with binder will have high mean MORs when fired at different temperatures.

5.6 CORE COMPOSITION [6] AND [7] DISCUSSION

Core composition [6] Figure 80 shows the mean setting time of these trials. Trial 4A has a mean setting time of twelve minutes. This is due to a higher percentage of plaster reacting with water. Trials 1A and 2A have twenty minutes mean setting time in the wooden core box. Trial 3A has fifteen minutes since it has a higher percentage of plaster than in Trials 2A and 1A. Trial 2D has fifteen minutes mean setting time, which is more than in Trial 4A since calcium silicate and plaster bond has a longer reaction time. Trials 1E and 2E have twenty minutes and twenty-five minutes respectively for mean setting since the latter is more dense (dried and treated).

All core trials showed high viscosity. Trial 2D has high viscosity, since plaster and calcium silicate with binder produce thicker slurry. The mixed slurry should have suitable viscosity depending on the application so that it can reach all corners of the mould or wooden core box. The mean setting times of composition [6] are good relative to ceramic cores produced in commercial⁽⁶⁾ industries. Composition [6] mean setting time suggests that use of binder will give a high slurry working life but it may be too viscous to fill the mould by gravity alone.

Core composition [7]: Figure 80 also shows the mean setting time of core trials mixed with water. In general, the mean setting times of all core trials were very low. The mixed slurry was very viscous and also had air bubbles when mixed. This is due to water crystallisation quickly bonding with plaster. Trials 1A and 2A had fifteen minutes of setting time in the wooden core box. Trial 3A had ten minutes and Trial 4A recorded eight minutes of mean setting time. Trial A core has reduced mean setting time with increase in plaster. Trial 2D had six minutes of mean setting time

since calcium silicate and plaster reaction bond quickly in comparison with Trials 1E and 2E, which had fifteen and ten minutes mean setting time respectively.

The pattern of mean setting times suggests that slurry mixed with water has very low viscosity and slurry-working life is short. This may have limitations in any commercial industry.

5.6.1 CORE COMPOSITION [6] AND [7] PROPERTIES

Core composition [6] Figure 81 shows the mean MORs of core trials at different fired temperatures. When removed from the wooden core box, cores were very strong with smooth surfaces; core trials recorded higher mean MORs when fired at elevated temperatures. This is due to the binder having resistance to elevated temperatures producing covalent bonds.

When fired at elevated temperatures, Trial A recorded high mean MORs. The higher percentage of plaster in these core trial results in higher mean MORs. Trial 4A gave a MOR of 5.78 MN/m^2 at $800 \text{ }^\circ\text{C}$. This suggests that a higher percentage of plaster and binder works as a bonding agent and will provide higher mean MORs when fired at elevated temperatures.

Trial 2D gave a higher mean MOR recorded than any other core trial in composition [6] when fired at elevated temperatures. This shows that the calcium present in core trials has a significant effect on the core strength. At $800 \text{ }^\circ\text{C}$, Trial 2D gave a MOR of 6.01 MN/m^2 . This suggests that calcium silicate and plaster with Ludox AM binder concentration gives greater strength to the ceramic cores.

Trial E cores have recorded higher mean MORs when fired at elevated temperature. Trial 2E has higher mean MORs than Trial 1E since the alpha plaster is denser. At 800 °C, Trials 1E and 2E gave MOR of 4.98 MN/m² and 5.05 MN/m² respectively.

CORE COMPOSITION [7]: Figure 82 shows the mean MORs of core trials when fired at different temperatures. When removed from the wooden core box, cores were very wet and sometimes broken, or could not be handled, if successfully removed from the wooden core box. The core composition [7] cores recorded very low mean MORs; the core trial uses water to bind. For completeness, the different MORs are considered, but in Trial A, higher percentage of plaster in core trials has also given low mean MORs. At 800 °C, Trials 1A and 4A have recorded MORs of 0.12 MN/m² and 0.24 MN/m² respectively.

Trials 2D also recorded lower mean MORs when fired at higher or lower temperatures. At 800 °C, Trial 2D has a MOR of 0.21 MN/m². This suggests that water crystallisation evaporates quickly and results in very dry and weak cores.

Trials 1E and 2E also recorded very low mean MORs when fired at elevated temperatures. The alpha plaster (Crystal R) gave lower mean MORs than beta plaster (Fine casting plaster). At 800 °C, Trial 2E gave a MOR of 0.22 MN/m² and Trial 1E a value of 0.28 MN/m². This suggests that high-density plaster loses water crystallisation more quickly.

5.6.2 REVIEW OF CORE COMPOSITION [6] AND [7]

Core composition [6] suggests that core trials record high mean MORs when fired at elevated temperatures. This suggests that using only binder to mix will result in high mean MOR but the core may have a rough surface after the firing process. Some commercial applications need a suitable mean MOR since it is difficult to remove the core from casting.

Core composition [7] core trials record very low mean MORs when fired at elevated temperatures. Manual handling is very difficult having a high impact on commercial applications, since cores may need to have adequate strength and surface finish to withstand the waxing process (applied for easy removal from mould).

Core compositions [6] and [7] were not evaluated in the casting process since composition [6] gave high strength, rough surface and composition [7] gave poor strength and manual handling. It is likely that water based cores may collapse once molten metal is poured. Castings with binder based ceramic cores may not leach with diluted acids or may need a pressurised auto-clave process.

Various binder systems now employed include binders for moulding compositions; inorganic⁽⁹²⁾ substances are the major components. However, inorganic binders have suffered from one or more deficiencies. The typical deficiencies exhibited by inorganic binders include the result that silicates suggested for molding shapes such as cores and moulds have poor collapsibility⁽⁹²⁾ and poor removal of the moulding shape from the metal casting.

Fused silica is used in most of the ceramic cores as a filler material due to its good thermal resistance. Since fused silica takes a longer time to leach out ⁽⁹³⁾ and magnesium oxide causes the mixed slurry to set too rapidly, an alternative for both materials is reviewed. Core compositions [8] and [9] replace the fused silica and magnesium oxide. Calcium carbonate is used as a filler material due to its nature of readily leaching with diluted acids and a good bonding agent with water. Tri-sodium citrates are emulsifying salts, which increase the slurry working life and mean setting time and find application as a binder in commercial food production; it is used to increase the shelf life and control slurry in the pasteurising process. Tri-sodium citrate is employed as a replacement for magnesium oxide to get changes in slurry setting time.

5.7 CORE COMPOSITION [8] AND [9] DISCUSSION

Core compositions [8] and [9] are reviewed, consisting of core trials with calcium carbonate and tri-sodium citrate. Core composition [8], using calcium carbonate as filler material, produced very low strength. Core composition [9], using tri-sodium citrate, gave a longer slurry working life.

Core composition [8]. Mixed slurry core trials had low viscosity since the cores were made with a large water to binder ratio. Figure 83 shows the mean setting time of composition [8]. Trial A recorded high mean setting times with a lower percentage of plaster. Trial 1A has thirty minutes and Trial 4A twenty minutes of mean setting time. The calcium carbonate bonds quickly with plaster. Trials 2A and 3A recorded twenty-five minutes of mean setting time. Trial 2D recorded a very low mean setting time since it would appear that calcium silicate, calcium carbonate and plaster bonds quickly, sometimes setting before a complete pour was possible. Trials 1E and 2E gave twenty minutes respectively.

Core composition [9]. Mixed slurry core trials also had low viscosity since cores are made with large parts of water to binder ratio. Figure 83 shows the setting time of core composition [9] on different core trials. The tri-sodium citrate has increased the setting rate in the wooden core box and reacted with water and binder to prolong the working of the mixed slurry. Trial 1A had thirty minutes and Trials 4A, 2D had forty minutes of mean setting time. Trials 2A and 3A recorded thirty and thirty-five minutes of mean setting time respectively. Trial 1E had thirty minutes and Trial 2E thirty-five minutes of mean setting time.

5.7.1 CORE COMPOSITION [8] AND [9] CORE PROPERTIES

Core composition [8]. Figure 84 shows the mean MORs of core composition [8] core trials when fired at different temperatures. When fired at elevated temperatures, cores have very low strength due to calcium carbonate decomposing at 800 °C. Trial A cores increased the strength with a higher percentage of plaster added to the core trial. At 200 °C, Trial 4A recorded 2.65 MN/m² as the highest mean MOR. Using calcium carbonate and firing then to elevated temperatures does have an extreme effect on core strength.

At 200 °C, Trial 2D gave a MOR of 3.03 MN/m². When fired at different temperatures, Trial 2D gave reduced strength because calcium carbonate acts to disrupt the bonding with other compounds. Since calcium carbonate has very low density, it reduces core weight when fired at elevated temperatures. Trial 1E and 2E gave high mean MORs when fired at 200 °C. At 200 °C, Trial 2E gave 2.85 MN/m² and Trial 1E 2.65 MN/m². Trials E also gave reduced mean MORs when fired at elevated temperatures.

Core composition [9]. These gave low mean MORs when fired at elevated temperatures. The change of magnesium oxide to tri-sodium citrate has no effect on the ceramic core strength. The mean setting of mixed slurry in the wooden core box has no effect on the strength of ceramic cores. Figure 85 shows the mean MORs of core composition [9] when fired at different temperatures.

When fired at 200 °C, a higher mean MOR is recorded on core Trial A. The plaster bonding with binder is high when fired at lower temperatures. The higher percentage

of plaster in core trials led to higher strength. Trial A cores have a reduced strength when fired at elevated temperatures.

Trial 2D recorded 3.25 MN/m^2 as a higher mean MOR when fired at low temperatures. This is due to the two-bonding system from calcium silicate and plaster. When fired at elevated temperature, Trial 2D reduced its mean MOR.

Trial E cores also have high mean MOR when fired at $200 \text{ }^\circ\text{C}$ and lose strength when fired at elevated fired temperatures. Trial 2E recorded higher mean MOR than Trial 1E since alpha plaster is denser than beta plaster. When fired at elevated temperatures, Trial 2E gave stronger MOR than Trial 1E.

5.7.2 CORE COMPOSITION [8] AND [9] REVIEW

The core composition [8] has been reviewed using a different filler material (calcium carbonate) and setting agent (tri-sodium citrate). These stages were not adopted for casting purposes. This is because visual observation showed that cores produced with calcium carbonate are very dry and light in weight. Consideration for manual handling is needed and casting temperatures of aluminium LM25 may break the core.

The use of calcium carbonate as a filler material is not recommended due to it decomposing at $800 \text{ }^\circ\text{C}$. This will have a higher effect on the core strength and integrity. Core removal with diluted citric and acetic acid is very easy, as calcium carbonate readily reacts with acids, but due to its density it is not suitable to use in the casting process. This confirms that fused silica is a better material to use as filler.

The use of tri-sodium citrate has increased the slurry working life of the mixed slurry in core trials. In cost terms, tri-sodium citrate is much more expensive than magnesium oxide. Tri-sodium citrate has no effect other than increasing the slurry working life. The core composition [9] may be used in commercial applications where longer slurry working life for making cores is needed. Composition [9] is not adopted for casting purposes since composition [9] similar to composition [5], cores will react with diluted acetic and citric acid. It is suggested that magnesium oxide is more suitable to use than tri-sodium citrate due to the latter's high cost.

6.0 CONCLUSIONS

6.1 Core Manufacture

- The overall aim of the project was to produce a water-based core for aluminium and light alloys capable of being leached out using dilute acid. This aim is achieved by using limited filler materials.
- The cores are largely water based using minimal binder. The cores also have suitable strength but there are limitations with manual handling in the “green” condition.
- Suitable leaching rates can be achieved using diluted citric and acetic acids.
- Cores can be manufactured simply and by a process amenable to scale up to commercial levels.
- A single cycle firing process following twenty four hours room temperature drying is the simplest and most feasible methodology.
- Cores can be produced with adequate “green” strength, using water based compositions.
- Depending on the specific application needed relative to an industrial process, cores can be produced using a wide range of compositions.
- Mechanical properties of fired cores (600 °C and 800 °C) are appropriate to commercial applications. Plain strain fracture toughness (0.087-0.14 MN m^{-3/2}) and Weibull parameters (>1) are consistent with this type of product and suggest the brittle factor of the cores. The critical flaw size (0.55-1.12 mm) suggests that core is enriched with high micro cracks.
- Cores have sufficient refractoriness to withstand molten aluminium and alloys.
- Cores made with a range of compositions can be readily leached using diluted acetic or citric acids in modest dilutions.

- Core removal process may require further jet washing for more complex shapes or to remove adhering layers at the internal surfaces of cast parts.
- Further process development is also needed to fine tune the removal process.

MATERIALS CONCLUSIONS

- **Plaster**, at 770 °C breaks down to calcium oxide with evolution of sulphur trioxide. This change of plaster when fired at elevated temperatures has direct effects when cores are produced using such materials.
- Higher additions of plaster in cores produce higher mean MOR when fired at 200 and 400 °C. The opposite results occur when cores are fired in the range 600 to 800 °C.
- High addition of plaster with less water and high binder causes mixed slurry to set very quickly.
- **Fused Silica** changes phase only when fired at 1300 °C. There is no effect when fired between 200- 800 °C. This precludes the possibility of generating harmful crystoballite
- The grain size of fused silica used (30/50 Mesh) produced cores with rough and uneven surface.
- Fused silica flour (-200 Mesh) produced a smooth core surface constituting an excellent filler material.
- **Magnesium oxide** is very refractory but has little effect on core properties after mixing.
- The higher additions of MgO produced quicker setting action when mixed with water and binder.

- No addition of MgO results in a very long setting action in core trials provided there is suitable addition of water and binder.
- **Calcium silicate** also caused quicker setting of the core and produced relatively high mean MOR ($>5 \text{ MN/m}^2$) with plaster addition.
- Addition of calcium silicate has very limited effect on the collapsibility of core after the casting process.
- **Lithium carbonate** compositions had little effect on mean MOR.
- **Binder**, (Ludox[®] AM) at higher additions in core trials increases the strength ($> 4.5 \text{ MN/m}^2$) with plaster when fired at more than $400 \text{ }^\circ\text{C}$.

6.2 COMPOSITION CONCLUSION

- **Core composition [1]**. Due to use of coarse fused silica (30/50 and -120), the uniform strength of core was not in balance. The higher percentage of magnesium oxide (1%) also made slurry-working life fast. The core removal was faster since high concentration of nitric was used. The composition of cores is not suitable for any commercial application
- **Core composition [2]**. Using the fine fused silica (-200) with coarse material also made no difference on the uniform strength of core. The magnesium oxide (0.1%) increased the slurry working life. Using much diluted acid (1:100, 1:1000) took more than twenty-four hours to attack the core. The composition of cores is not suitable for any commercial application.
- **Core composition [3]**. Using the fine fused silica (-200 and -120) made the core surface smooth and better packing of materials used. The strength of cores also increased to 20%. The core removal was easy in dilute (1:1 and 1:5)

acetic and citric acid. The cores can be used in industry provided there is low firing temperature on cores.

- **Core composition [4].** Cores were produced without any additives which show that cores have long slurry working life. The cores were fired at higher temperature (with less contraction on diameter and length) suggesting the commercial application where high firing is required on cores.
- **Core composition [5].** Adding the magnesium oxide has made the core production faster with standardised dimensions. The firing also gave less contraction on diameter and length. The mechanical properties suggest that cores have high probability of failure. Further work is required to prove the mechanical properties. The commercial application is recommended with fine tune of mechanical properties.
- **Core composition [6]and [7].** Cores made with water risk in manual handling and weak in MOR after firing. Cores made with binder have high MOR but rough surface with probability of no removal through acid due to bonding system. The commercial application is not recommended for such type of cores.
- **Core composition [8] and [9].** Changing the filler material to calcium carbonate suggests cores have very low density, breaking when fired. The tri-sodium citrate increases the slurry working life but is costly.

7.0 SUGGESTIONS FOR FURTHER WORK

- **Calculating porosity, density and leaching rate on core trials.** This will help in finding the density effect on the leaching rate. This is very useful since the casting industry utilises complex shapes with highly dense silica cores.
- **Phase change analysis at elevated temperatures on different core trials.** The silica cores do not change phase when fired at less than 800 °C. Adding other materials to silica, may cause a dimensional effect when fired at less than 400 °C. This would need to be known if such cores were to be utilised in a commercial process such that dimensional changes could be accommodated.
- **The commercial production and test for core composition [4] and [5].** This will test the strength, dimensional control and leaching of silica cores with different low melting alloys. The specific requirement of commercial application can be met by using specific core trials with varying strength and dimensional attributions.
- **Using finer flour grade of fused silica as filling material.** This will give a smoother finish, higher strength and improved leaching from diluted acids.
- **Creep Characteristics.** This will enable an understanding to be gained of the stress deformation at elevated fired temperatures. The high temperature (> 1000 °C) compressive creep tests give a creep deformation as a function of temperature and stress level.

- **Changes in binder grades.** (Ludox HS and SK). This will have an effect on slurry setting, manual handling and strength with different core trials. In this research binder (LudoxAM) was not changed.

- **Injection Process of mixed slurry.** This is helpful in achieving the higher packing of particles leading to higher MOR in core trials. This investigation may need high financial investment.

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