

RELATIONSHIPS BETWEEN CHEMICAL, PHYSICAL AND MINERALOGICAL CHARACTERISTICS OF THE RUPELIAN BOOM CLAY, BELGIUM

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ABSTRACT: The Boom Clay is an important raw material for the Belgian structural clay products industry. It can be divided into several lithological sub-units. In order to obtain an insight in the parameters which characterize these different units, and which affect the overall properties of the clay, a detailed chemical, physical and mineralogical analysis was carried out. Pearson correlation coefficients were calculated between the variables, which were then placed in four correlation groups: a quartz/clay mineral group, a reducing component group, a carbonate group and a feldspar group. This treatment of the data allowed three analysis parameters to be selected: $<2 \mu\text{m}$ fraction, organic carbon and CO_2 . The other variables could be calculated fairly accurately from these by means of regression lines.

INTRODUCTION

The Boom Clay is used extensively as a raw material by the Belgian brick and bloating clay industries and this paper presents the results of a detailed chemical, physical and mineralogical investigation aimed at determining the parameters which are important for these applications. The type outcrops of the Boom Clay are the excavations for structural clay product manufacture in the cuesta front just to the north of the River Rupel. These occupy a zone 5–15 km wide over a distance of ~65 km. The clay unit is of Middle Oligocene (=Rupelian) age and its stratigraphy and sedimentology have recently been investigated by Vandenberghe (1978), who divided it into several sub-units. In the north-west European sedimentary basin the Boom Clay is considered on lithological and geometrical criteria to be equivalent to the Dutch, German and Polish 'Septarienton' and to the Danish Branden Clay. The exact chronostratigraphic position of the different lithological sub-units has not yet been established precisely. It is probable that they partly represent lateral facies variations at a particular time level. In Belgium, a sub-division of the Boom Clay into a lower grey clay (the Land van Waas Clay) and an upper black clay (the Putte Clay) has been tentatively proposed (Vandenberghe, 1978).

The Boom Clay is a marine shelf deposit of very widespread occurrence. Silty clay and heavy clay layers of some tens of centimetres regularly alternate. Besides these vertical grain-size variations, layers rich in organic matter and carbonate also occur; septaria are mostly developed in the carbonate-rich layers. Pyrite frequently occurs as concretions up to 10 cm across. The sequence of these different layers is constant over the whole of the

present area of exploitation. The total thickness of the Boom Clay in this area varies between 10 and 50 m.

MATERIALS AND METHODS

In order to cover the whole thickness of the clay formation, outcrops in three clay pits (Sint-Niklaas (SN), Terhagen (TH) and Kruibekke (KR)) were channel-sampled. Two samples (SNPR and THPR) were also taken after extrusion of the mixed bulk clay. In all, 21 samples were examined and their positions in relation to stratigraphic levels within the Boom Clay are shown in Fig. 1.

For major-element determinations, the powdered sample was dissolved by a LiBO_2 -fusion method (Suhr & Ingamells, 1966). SiO_2 , Al_2O_3 , CaO and P_2O_5 were determined by emission spectrometry in an Ar-plasma. Fe_2O_3 , MgO , Na_2O , K_2O , MnO and TiO_2 were determined by atomic absorption spectrometry in an air/acetylene mixture.

Adsorbed water (H_2O^-) and loss on ignition were determined by heating at 105°C and at 1000°C , respectively. The total water content (H_2O^\pm) was determined with an evolved moisture analyser. FeO was determined titrimetrically (Shafer, 1966). Fluorine was

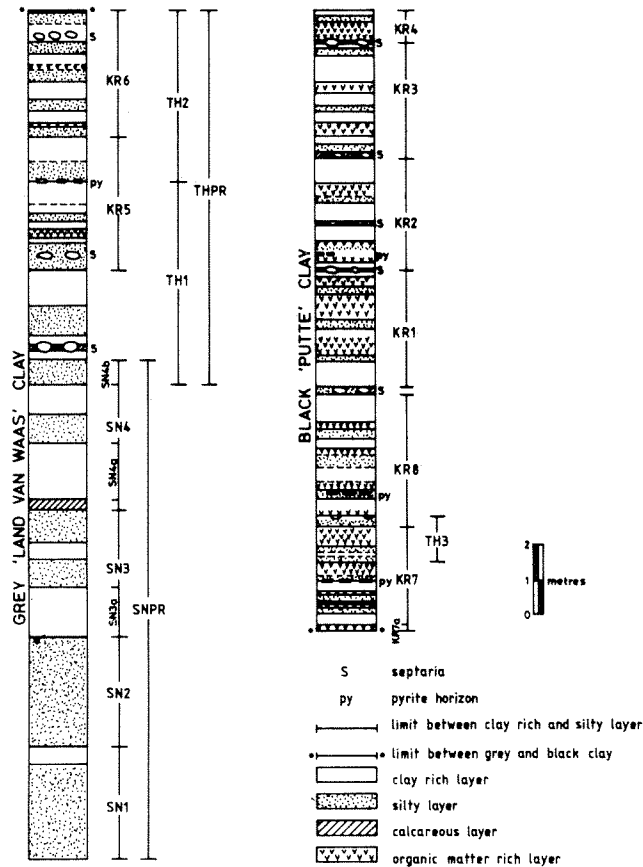


FIG. 1. Summary section of the Boom Clay.

determined with a specific-ion electrode (Josephson *et al.*, 1977). Total carbon and sulphur were analysed by combustion methods at 1100°C and 1350°C, respectively. Carbonate contents were determined from the CO₂ evolved on HCl treatment. Organic carbon was determined with the Walkley-Black method (Allison, 1965). Water-soluble sulphates were determined by turbidimetry (Bennett & Reed, 1971; Patterson & Pappenhagen, 1978).

Grain-size distributions were determined by a combination of wet sieving (>38 μm) and sedimentation (38–2 μm). The Atterberg plastic and liquid limits were determined following the procedure of Sowers (1965). The Pfefferkorn plasticity number (at 15 mm residual depth) was determined after van der Velden (1979). Total (external and internal) specific surfaces were measured using a water adsorption method (van der Velden, 1978).

Quartz, microcline and plagioclase were quantitatively determined by X-ray diffractometry (Weber *et al.*, 1973). The calcite content was calculated from the CO₂ content. The pyrite content was calculated from the total sulphur value after subtracting an amount of sulphur equivalent to the water-soluble sulphate. The theoretical clay mineral percentage was calculated from the equilibrium moisture content (van der Velden, 1978) and the H₂O⁻ content after the (modified) method of Keeling (1962).

Semi-quantitative estimations of each clay mineral species in the <2 μm fraction were made from the diffraction traces of the glycol-treated slide mounts. Areas under the basal peaks were measured in preference to peak heights. Illite was used as an internal standard while the areas of the smectite and kaolinite peaks were divided by 3 (Vandenberghe, 1978); values so obtained were normalized to 100%. Amounts of each clay species in the bulk sample were obtained by multiplying its percentage in the <2 μm fraction by the factor $x/100$, where x represented the percentage of <2 μm fraction in the bulk sample. This was considered justified because for each bulk sample the <2 μm fraction was mainly composed of clay minerals and the sum of the quartz, microcline, plagioclase, pyrite, calcite and <2 μm percentages was close to 100%.

RESULTS

Table 1 gives the mean percentages (\bar{x}), the lower and upper limits, and standard deviations (s) of the different chemical and physical variables and of the mineralogical components. There are considerable variations in the results for particular parameters due to the presence of specific layers rich in silt or clay, or in organic matter or carbonate. During excavation, these different layers are thoroughly mixed so that they do not cause problems during the shaping, drying and firing processes.

DISCUSSION AND IMPLICATIONS

Statistical treatment of the data was necessary in order to obtain an insight into the relationships between the variables, and to select the appropriate parameters for evaluation of the clay.

Structure of the analytical data

In the first instance, Pearson correlation coefficients (r) were calculated for the variables, the computer programs of Nie *et al.* (1975) being used. Only the most interesting correlations are given in Table 2. On plotting the scatter diagrams, the data were seen to be

TABLE 1. Summary of chemical, physical and mineralogical results. Values in % except for F in ppm and total specific surface in m²/g.

	$\bar{x} = \frac{\Sigma x}{n}$ n = 21	min-max	$s = \sqrt{\frac{\Sigma(x - \bar{x})^2}{n - 1}}$
SiO ₂	62.65	54.19-73.38	5.75
Al ₂ O ₃	14.61	8.65-18.41	2.80
Fe ₂ O ₃	3.35	2.18-4.42	0.57
FeO	1.33	0.89-1.76	0.28
MgO	1.19	0.94-1.34	0.12
CaO	1.36	0.66-3.50	0.81
Na ₂ O	0.42	0.33-0.50	0.06
K ₂ O	3.07	2.55-3.31	0.24
TiO ₂	0.83	0.59-0.99	0.13
P ₂ O ₅	0.09	0.08-0.11	0.01
H ₂ O ⁻	5.85	3.80-7.60	0.86
H ₂ O ⁺	1.77	0.90-2.55	0.46
Loss on ignition	7.95	3.77-11.38	1.97
S _{tot}	1.00	0.46-1.98	0.42
SO ₄ ²⁻	0.49	0.22-1.41	0.33
C _{tot}	1.79	0.69-4.56	0.87
Organic C	1.66	0.64-4.50	0.90
CO ₂	0.38	0.00-1.87	0.53
F	558	334-737	119
<2 μm	49.6	23.5-63.3	10.8
<10 μm	63.8	29.6-82.5	14.4
<20 μm	70.1	32.7-89.0	15.0
>63 μm	6.1	0.5-33.6	9.0
>106 μm	0.9	0.1-4.1	1.0
Plastic limit	25.2	18.4-32.3	4.0
Liquid limit	51.0	30.9-63.0	8.3
Plasticity index	25.9	9.4-38.0	6.8
Pfefferkorn plasticity no.	44.5	29.7-60.2	8.0
Total specific surface	140	76-184	27
Quartz	35.2	23.8-58.3	10.0
Microcline	8.8	6.5-11.3	1.3
Plagioclase	4.5	3.2-6.2	0.7
Pyrite	1.5	0.7-2.5	0.5
Calcite	0.9	0.0-4.3	1.2
Smectite	33	19-42	7
Illite	12	3-23	5
Kaolinite	5	1-9	2
Theoretical clay mineral content	56	37-71	8

regularly spaced over the entire range, indicating that the correlation coefficients were valid.

SiO₂ is negatively correlated with all the chemical constituents of the clay minerals. S_{tot} and organic carbon reflect the reducing circumstances obtaining during sedimentation of the clay. TiO₂ shows a positive correlation with Al₂O₃ ($r = 0.88$) and with <2 μm ($r = 0.80$), indicating a tendency for TiO₂ to be concentrated in the fine particle fraction,

probably as anatase. F is also positively correlated with Al_2O_3 ($r = 0.84$) and with $<2 \mu\text{m}$ ($r = 0.88$), which is an indication that F is present as a substitution anion. CaO shows a high positive correlation only with CO_2 ($r = 0.98$), which demonstrates that most of the calcium occurs as a carbonate. MnO, Na_2O , P_2O_5 and SO_4^{2-} show no correlations; they are also present in small amounts.

As expected, the $<2 \mu\text{m}$ fraction is negatively correlated with SiO_2 and positively correlated with the clay mineral components. The $>63 \mu\text{m}$ fraction is mainly composed of quartz ($r = 0.86$), which is also shown in the positive correlation with SiO_2 ($r = 0.96$), and in the negative correlations with the clay mineral constituents. Plasticity is mainly determined by the clay mineral content, a fact confirmed by the mutual correlations. The Atterberg plasticity index shows no correlations, in contrast to the plastic and liquid limits. The Pfefferkorn plasticity number, however, gives much higher correlations, indicating that this test is better related to the clay content. Therefore, and because the Pfefferkorn impact test is also more precise, quicker and easier to carry out than the Atterberg method, this test is recommended. The total specific surface is mainly determined by the clay minerals, hence the mutual relationship.

Quartz is negatively correlated with the clay minerals, while pyrite is positively correlated with these. The reducing sedimentation conditions were conducive to pyrite formation. The theoretical clay mineral percentage shows interesting correlations with several chemical and physical variables.

The good correlations of the three main clay minerals with the other variables are an indication of: (i) the presence of certain elements in a specific clay mineral; (ii) the grain-size control on the clay mineralogy; (iii) the influence of these minerals on the plasticity and total specific surface; (iv) the validity of the quantification method, taking into account the semi-quantitative nature of the results. These statements can best be illustrated by the most important clay mineral in the Boom Clay, i.e. smectite.

The statistical treatment indicated that the smectite in the Boom Clay must be Fe^{3+} -rich. In order to demonstrate this, several parameters which depend solely on the clay content were derived and correlations calculated. The derived variables were:

$$\% \text{ rest SiO}_2 = \% \text{ SiO}_2 - \% (\text{free SiO}_2 + \text{SiO}_{2\text{micro}} + \text{SiO}_{2\text{plagio}});$$

$$\% \text{ rest Al}_2\text{O}_3 = \% \text{ Al}_2\text{O}_3 - \% (\text{Al}_2\text{O}_{3\text{micro}} + \text{Al}_2\text{O}_{3\text{plagio}});$$

$$\% \text{ rest FeO} = \% \text{ FeO} - \% \text{ FeO}_{\text{pyrite}};$$

$$\% \text{ rest Fe}_2\text{O}_3 = \% \text{ Fe}_2\text{O}_3 + 1.11 \% \text{ rest FeO}.$$

The significant correlations are given in Table 3. There is a high positive correlation ($r = 0.88$) between rest Fe_2O_3 and smectite (for comparison: $r(\text{rest Fe}_2\text{O}_3\text{-illite}) = 0.55$ and $r(\text{rest Fe}_2\text{O}_3\text{-kaolinite}) = 0.64$), as well as between rest Fe_2O_3 and rest SiO_2 ($r = 0.84$) and rest Al_2O_3 ($r = 0.85$). This mutual relationship between Al, Si and Fe confirms that these elements are the main constituents of the smectite. The smectite particles occur most frequently in the $<2 \mu\text{m}$ fraction ($r = 0.82$) and it is also well-known they have the largest total specific surface ($r = 0.90$). It therefore follows that rest Fe_2O_3 is highly correlated with $<2 \mu\text{m}$ and total specific surface ($r = 0.91$ for both). Si and Al are also correlated with these two physical parameters.

In summary, good mutual correlations are found between:

(i) the chemical variables which are mainly determined by the ratio quartz/clay minerals;

TABLE 2. Correlation matrix ($n = 21$); correlations significant above 0.63 (99.9% confidence level) and above 0.55 (99.5% confidence level)

	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	FeO	MgO	K ₂ O	H ₂ O ⁺	H ₂ O ⁻	Loss on ignition	S _{tot}
SiO ₂	1	-0.96	-0.79	-0.68	-0.85	-0.77	-0.86	-0.82	-0.96	-0.76
Al ₂ O ₃		1	0.67	0.80	0.87	0.87	0.80	0.81	0.88	0.69
Fe ₂ O ₃			1	0.24	0.52	0.47	0.78	0.64	0.79	0.77
FeO				1	0.71	0.75	0.36	0.61	0.60	0.49
MgO					1	0.80	0.69	0.52	0.74	0.42
K ₂ O						1	0.62	0.61	0.60	0.43
H ₂ O ⁺							1	0.68	0.80	0.63
H ₂ O ⁻								1	0.83	0.79
Loss on ignition									1	0.84
S _{tot}										1
Organic C										
<2 μm										
>63 μm										
Plastic limit										
Liquid limit										
Pfefferkorn plasticity no.										
Total specific surface										
Quartz										
Pyrite										
Theoretical clay mineral content										
Smectite										
Illite										
Kaolinite										

TABLE 3. Correlations with smectite.

	Rest Al ₂ O ₃	Rest Fe ₂ O ₃	Rest SiO ₂	<2 μm	Total specific surface	Smectite
Rest Al ₂ O ₃	1	0.85	0.84	0.96	0.87	0.70
Rest Fe ₂ O ₃		1	0.84	0.91	0.91	0.88
Rest SiO ₂			1	0.84	0.77	0.68
<2 μm				1	0.95	0.82
Total specific surface					1	0.90
Smectite						1

(ii) the physical parameters such as the <2 μm fraction, the Pfefferkorn plasticity number and the total specific surface, which in turn are highly correlated with the clay mineral content;

(iii) the reduced phases, i.e. pyrite and organic carbon.

The carbonate and feldspar contents (microcline and plagioclase) vary independently.

These conclusions are illustrated in Fig. 2 where the mutually correlated parameters are grouped. It must be stressed that the relationships between these groups have not the same significance. There is a causal link between the physical parameters and the clay minerals, whereas the relation between the latter and the reducing components is mainly sedimentological.

Organic C	<2 μm	>63 μm	Plastic limit	Liquid limit	Pfefferkorn plasticity no.	Total specific surface	Quartz	Pyrite	Theoretical clay mineral content	Smectite	Illite	Kaolinite
-0.70	-0.95	0.81	-0.87	-0.75	-0.91	-0.89	0.96	-0.77	-0.93	-0.70	-0.78	-0.81
0.61	0.95	-0.82	0.85	0.76	0.91	0.85	-0.95	0.70	0.91	0.68	0.82	0.80
0.60	0.79	-0.57	0.77	0.54	0.64	0.85	-0.78	0.71	0.79	0.77	0.48	0.52
0.49	0.67	-0.57	0.55	0.62	0.76	0.46	-0.66	0.60	0.60	0.31	0.77	0.67
0.43	0.87	-0.87	0.69	0.89	0.85	0.77	-0.84	0.48	0.71	0.63	0.72	0.76
0.40	0.85	-0.83	0.74	0.72	0.83	0.74	-0.81	0.43	0.74	0.68	0.70	0.58
0.50	0.84	-0.67	0.83	0.66	0.71	0.92	-0.82	0.55	0.86	0.79	0.49	0.67
0.69	0.75	-0.56	0.73	0.46	0.73	0.69	-0.78	0.77	0.92	0.45	0.72	0.66
0.80	0.86	-0.67	0.84	0.62	0.86	0.80	-0.91	0.86	0.89	0.56	0.76	0.80
0.87	0.67	-0.35	0.83	0.33	0.70	0.63	-0.68	0.95	0.76	0.43	0.64	0.53
1	0.59	-0.32	0.74	0.38	0.75	0.52	-0.60	0.90	0.65	0.27	0.71	0.45
	1	-0.85	0.85	0.85	0.91	0.95	-0.95	0.67	0.93	0.82	0.78	0.72
		1	-0.64	-0.79	-0.76	-0.77	0.86	-0.39	-0.73	-0.73	-0.61	-0.64
			1	0.58	0.83	0.84	-0.84	0.77	0.84	0.65	0.68	0.67
				1	0.79	0.79	-0.78	0.41	0.68	0.71	0.64	0.63
					1	0.80	-0.90	0.73	0.83	0.61	0.85	0.70
						1	-0.88	0.57	0.91	0.90	0.59	0.66
							1	-0.69	-0.91	-0.72	-0.77	-0.81
								1	0.72	0.36	0.71	0.58
									1	0.72	0.74	0.72
										1	0.30	0.38
											1	0.65
												1

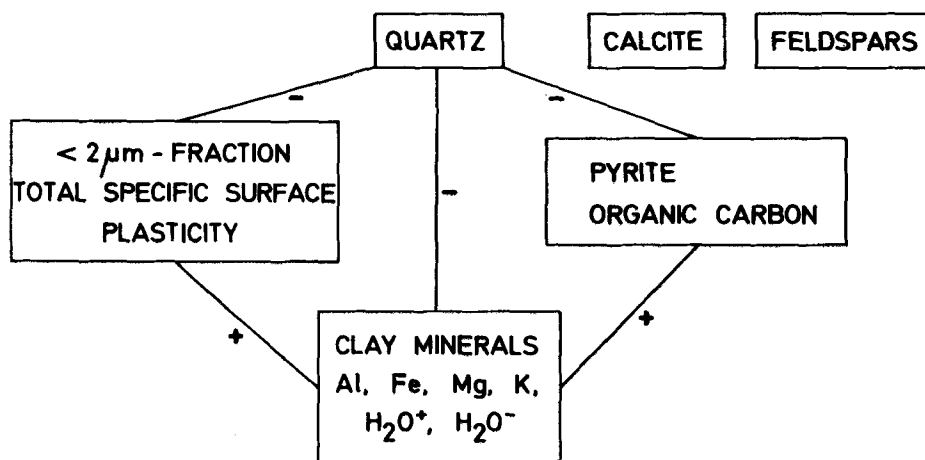


FIG. 2. Correlation groups.

Implications

This statistical analysis allows a grouping of the different variables and indicates a limited set of parameters which would be of use in characterizing the clay for its ceramic end-uses. By selecting some parameters, the others can be calculated fairly accurately. The following parameters are proposed.

<2 μm fraction. This has been selected because it gives high correlation coefficients with many variables and can be determined quickly and precisely in a structural clay product laboratory. Least-squares regression lines were calculated between the variables which gave correlation coefficients >0.90 with $<2 \mu\text{m}$. Thus when the $<2 \mu\text{m}$ fraction is determined, the other variables can be calculated. From these calculated variables, others can be derived for which the correlation coefficients are also >0.90 . The regression lines are given in Table 4, together with the correlation coefficients and the standard estimation errors ($s_{y,x}$). The standard estimation error is simply the standard deviation of the analysed values from the calculated values and is a measure of the scatter about the regression line. As can be seen, the calculated values correspond very well with the analysed data, the deviation being in most cases $<3\%$.

Pyrite and organic carbon. A high correlation exists between the pyrite and organic carbon contents ($r = 0.90$). By means of the regression line one variable can be calculated from the other: e.g. pyrite = $0.65 + 0.51$ organic carbon ($s_{y,x} = 0.2$).

Carbonates. CaO and calcite are highly correlated ($r = 0.98$). After CO_2 content determination, the total calcium content can be calculated from the regression line: CaO = $0.79 + 1.50 \text{CO}_2$ ($s_{y,x} = 0.17$).

Feldspars. The total feldspar content in the Boom Clay varies between 9.8 and 15.7% ($\bar{x} = 13.3\%$). Feldspars act as fluxes during the firing of ceramic ware but differences in amount in the Boom Clay are not wide enough to cause any significant variations in firing behaviour. Therefore, and also due to difficulties in quantifying feldspar minerals, feldspar contents are not used as a routine control.

TABLE 4. Regression lines, $s_{y,x}$, and r values with

	Regression line	$s_{y,x}$	r
	$s_{y,x} = \sqrt{\frac{(y_{\text{calc}} - y_{\text{anal}})^2}{n}}$		
SiO_2	= $87.67 - 0.50 <2 \mu\text{m}$	1.74	-0.95
Al_2O_3	= $2.42 - 0.25 <2 \mu\text{m}$	0.86	0.95
Rest Al_2O_3	= $0.69 + 0.26 <2 \mu\text{m}$	0.86	0.96
Total Fe_2O_3	= $1.84 + 0.06 <2 \mu\text{m}$	1.02	0.93
Rest Fe_2O_3	= $1.93 + 0.04 <2 \mu\text{m}$	0.20	0.91
$<10 \mu\text{m}$	= $-1.60 + 1.32 <2 \mu\text{m}$	1.6	0.99
$>20 \mu\text{m}$	= $100.75 - 1.43 <2 \mu\text{m}$	2.4	-0.98
Pfefferkorn plasticity no.	= $11.25 + 0.67 <2 \mu\text{m}$	3.3	0.91
Total specific surface	= $21.61 + 2.39 <2 \mu\text{m}$	8	0.95
Quartz	= $78.72 - 0.88 <2 \mu\text{m}$	2.9	-0.95
Theoretical clay mineral content	= $23.97 + 0.65 <2 \mu\text{m}$	3	0.93
Loss on ignition	= $28.65 - 0.33 \text{SiO}_2$	1.00	-0.96
Fe_2O_3	= $-0.17 + 0.73 \text{total Fe}_2\text{O}_3$	0.34	0.91
H_2O^+	= $-0.42 + 0.02 \text{total specific surface}$	0.67	0.92
Rest SiO_2	= $36.37 - 0.50 \text{quartz}$	2.87	-0.93
H_2O^\pm	= $-1.21 + 0.16 \text{theoretical clay mineral content}$	0.66	0.97
H_2O^-	= $-0.05 + 0.11 \text{theoretical clay mineral content}$	1.18	0.92
Smectite	= $2.60 + 0.21 \text{total specific surface}$	4	0.90

CONCLUSIONS

A detailed chemical, physical and mineralogical investigation was undertaken to evaluate the Boom Clay as a raw material for the structural clay product industry. Significant variations have been found in composition and properties of the different lithological units. Statistical analysis of the results has revealed mutual relationships between the variables which characterize the clay. These variables can be placed in four correlation groups: a quartz/clay mineral group, a reducing component group, a carbonate group and a feldspar group. This treatment of the data has allowed selection of three parameters to be analysed: $<2 \mu\text{m}$ fraction, organic carbon content and CO_2 content. The other variables can be fairly accurately calculated by means of least-squares regression lines.

This approach justifies the use of only a limited number of analyses to characterize the clay, and can be applied directly in the control of the raw material for the manufacture of bricks and bloated clay products.

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RESUME: L'argile de Boom est une matière première importante pour l'industrie des briques et des argiles gonflantes en Belgique. Ce dépôt d'argile peut être subdivisé en plusieurs couches. On a effectué des analyses détaillées des propriétés chimiques, physiques et minéralogiques en vue d'obtenir des indications sur les paramètres qui caractérisent ces différentes couches lithologiques ainsi que de ceux qui sont importants en vue des applications. On calcule des coefficients

de corrélation de Pearson entre les variables. Ceux-ci peuvent être classés en 4 groupes: quartz/argile, matières réductrices, carbonates et feldspaths. Le traitement des données permet de sélectionner trois paramètres qui doivent être analysés: la fraction $<2 \mu\text{m}$, le carbone organique et le CO_2 . Les autres variables peuvent aisément être calculés avec précision au moyen de courbes de régression obtenues par moindres carrés.

KURZREFERAT: Boom-Ton (Rupelton) ist ein bedeutender Rohstoff für die belgische Baustoffindustrie. Er kann in verschiedene Einheiten untergliedert werden. Zur Kennzeichnung der Eigenschaften dieser unterschiedlichen lithologischen Einheiten wurden chemischen, physikalische und mineralogische Analysen durchgeführt, deren Ergebnisse statistisch verrechnet wurden. Anhand der Pearson-Korrelationskoeffizienten wurden vier Gruppen unterschieden: Quarz/Tonmineral, reduzierende Komponenten, Carbonate und Feldspäte. Die für die Unterscheidung der Gruppen wichtigsten Parameter waren der Gehalt an Korngrößen $<2 \mu\text{m}$, an organischem Kohlenstoff und an CO_2 . Daraus konnten die übrigen analytischen Kenngrößen durch Regression mit leidlicher Genauigkeit berechnet werden.

RESUMEN: La arcilla es una materia prima importante para la industria belga de productos arcillosos. La formación arcillosa de Boom puede dividirse en varias subunidades litológicas. Para conocer los parámetros que caracterizan las diferentes unidades litológicas y cuáles son los que afectan al conjunto de las propiedades de la arcilla, se han llevado a cabo detallados análisis químicos, físicos y mineralógicos. Se han calculado los coeficientes de correlación de Pearson entre variables, lo cual permite clasificarlas en cuatro grupos de correlación: un grupo cuarzo/mineral de la arcilla, un grupo de componentes reductores, un grupo de carbonatos y uno de feldspatos. Este tratamiento de los datos ha permitido hacer una selección de tres parámetros analíticos: fracción $<2 \mu\text{m}$, carbono orgánico y CO_2 . A partir de estos parámetros, las otras variables pueden determinarse con bastante precisión, mediante regresión lineal.