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AN INVESTIGATION INTO THE CORRELATION BETWEEN DIFFERENT SURFACE AREA DETERMINATION TECHNIQUES APPLIED TO VARIOUS LIMESTONE-RELATED COMPOUNDS

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ABSTRACT

This communication reports an investigation between various methods used to measure the surface area of ground materials used in the cement industry. From the results is seems possible to make a reasonable estimate of the limestone, lime and gypsum BET surface areas from a Blaine surface area determination. *Copyright © 1996 Elsevier Science Ltd*

Introduction

Surface area is an important property of powders. It is a very useful indication of the reactivity of a particular material, and it plays a particularly important role in the cement industry where it is used as one of the control parameters in the production process.

In the cement industry the usual method of surface area determination is the so-called Blaine method. This method measures the time for a specific volume of air to flow through a known volume of compacted powder and together with the density of the substance, this is used to calculate the specific surface area of the sample. The main advantages of this technique are that it is simple and rapid. However, it is not very accurate and suffers from a number of weaknesses, e.g. variable particle shape and bed tortuosity[1] and becomes extremely unreliable at surface areas greater than 500 m^2/kg[2-3]. In situations where accurate measurements are required, one of the most common methods to measure surface area is the BET (Brunauer, Emmett and Teller) method. This method relies on a mathematical formula that describes the adsorption of a particular gas on the finely divided material to calculate its surface area[4] and measures both the internal and the external surface area of a material. However, it requires a skilled operator to perform it properly and is furthermore time consuming.

The PPC group of companies operates several limestone quarries. The limestone is used mainly as a raw material for cement manufacture, but also for the production of synthetic gypsum, and lime (after calcining). It was decided to use these three types of material originating from the same limestone deposit at Slurry in the North West Province of South

TABLE 1
Chemical Analysis of Material Used in the Investigation

Constituents	Limestone	Lime	Gypsum
SiO_2	4.4	7.1	2.1
Al_2O_3	0.5	1.7	0.3
Fe_2O_3	0.5	0.9	0.4
Mn_2O_3	0.1	0.2	0.1
TiO_2	<0.1	0.1	0.1
CaO	51.5	84.7	32.9
MgO	0.8	1.3	2.3
P_2O_5	<0.1	<0.1	<0.1
SO_3	<0.1	0.1	47.1
Cl	0.0	0.0	0.0
K_2O	0.1	0.1	0.1
Na_2O	0.1	0.3	0.2
LOI	41.7	3.4	14.5
TOTAL	99.7	99.9	100.1

Africa, and investigate the possible relationship between the Blaine and BET surface area measurements. The aim was to obtain, with a fast and easy Blaine measurement, an indication of the BET surface area of the sample. A number of limestone and imestone-derived compounds was therefore investigated with both techniques and the results are described in this note.

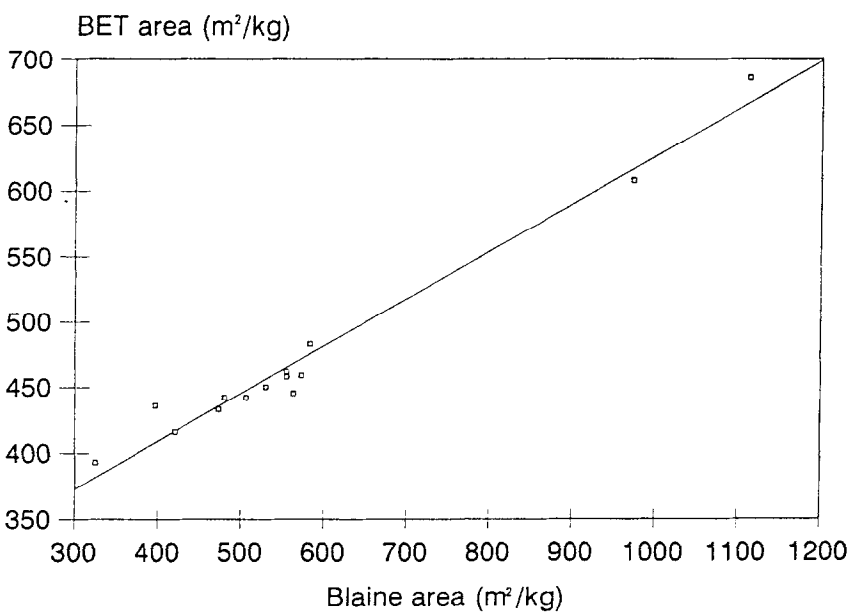

FIG. 1.
BET vs Blaine surface area for ground limestone.

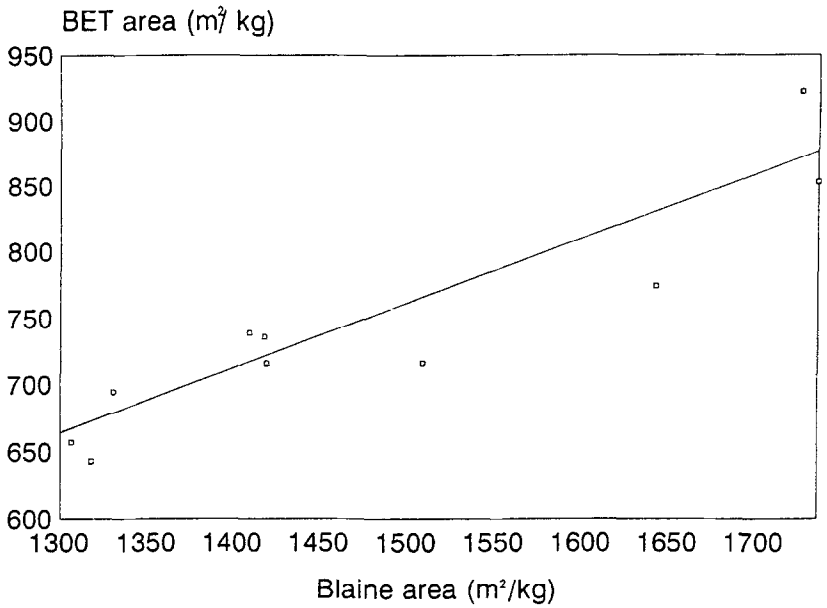

FIG. 2.
BET vs Blaine surface area for a ground lime.

Experimental Procedure

Limestone was dry ground in a ring mill for different periods of time. Some of the limestone was calcined at 1000°C for 1 hour to produce CaO which was also ground for various time periods. imestone was reacted with H_2SO_4 to produce synthetic gypsum which was treated in a similar manner. Each sample was then split into two parts, one of which was then measured according to standard procedures to determine its Blaine - and the other its BET surface area. In using the Blaine surface area measurement method, the true density of each individual sample was determined and a constant orosity was assumed for all the samples of all the materials and used in the calculation of the Blaine value. A more detailed description of the computation formula can be found in [5]. The chemical nalysis for each type of material is summarised in Table 1, while Figures 1-4 display graphically the relationship between BET and Blaine surface areas.

Results and Discussion

When evaluating the results, the accuracy level of each technique should be clearly borne in mind. The accepted accuracy of the Blaine method is lower than that of the BET method[2,3]. From the results, it appears that the lime obtained from calcining the limestone had the highest surface area after grinding. This is not surprising, since lime is generally soft and easily ground. The ground limestone and gypsum have similar surface areas after

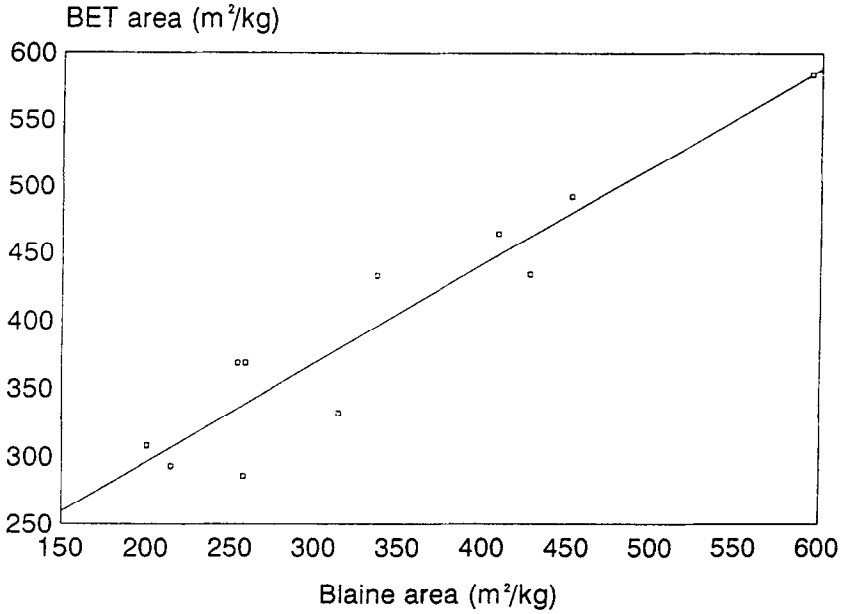

FIG. 3.
BET vs Blaine surface area for ground gypsum ($CaSO_4 \cdot 2H_2O$).

grinding, which are lower than those of the lime. The chemical analyses of the various materials summarised in Table 1 correspond to typical values for these materials.

In all three figures Figures 1-3 there is a reasonable straight line relationship between the Blaine- and BET surface areas of the respective materials. Linear regressions using a least

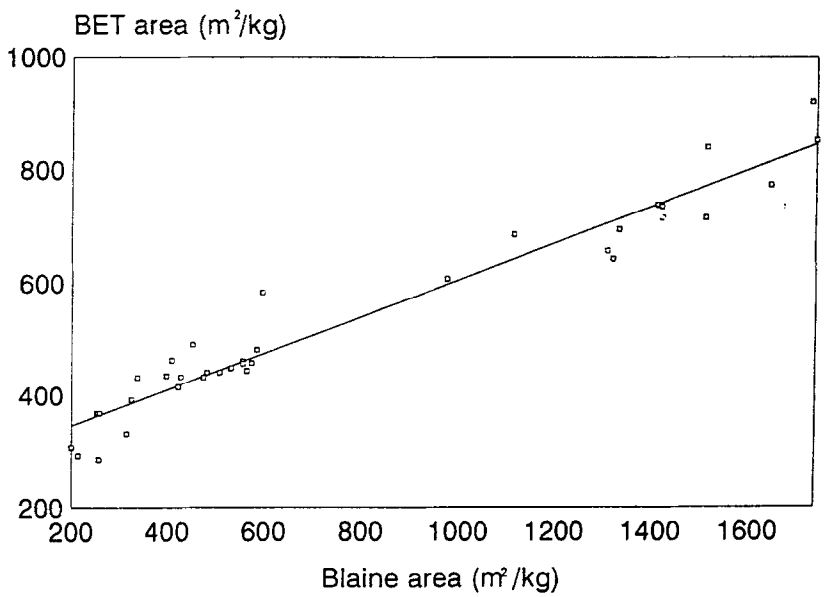

FIG. 4.
BET vs Blaine surface area for ground limestone, lime and gypsum.

TABLE 2
Correlation Coefficients And Constants Obtained By Fitting the Data for the Different Types of Material to an Equation of the Form Y = Mx + CUsing a Least Squares Difference Method (Standard Deviations are Given in Brackets)

Material	m	c	Correlation coefficient
Limestone	$3.62x10^{-1}$(±0.16)	264.1(±13)	0.986
Lime	$4.87x10^{-1}$(±0.90)	29.5(±43)	0.884
Gypsum	$7.31x10^{-1}$±0.87)	149.9(±33)	0.942
All three above	$3.26x10^{-1}$(±0.14)	281.2(±42)	0.969

y = BET surface area
x = Blaine

squares difference technique were done for all the data and the correlation coefficients and constants obtained are summarised in Table 2.

The best fit is obtained in the case of the limestone, while there is more scatter in the data for the other two materials, lime and gypsum. This scatter might be due to the nature of the lime and gypsum, which are both much softer than limestone and therefore yield more variation in the particle size when it is ground. The graph for the gypsum can still be used to get a reasonable estimate of the BET surface area from a Blaine measurement, but the lime graph is of no practical use.

When all the results are presented in one graph (Figure 4), one finds that the overall fit is quite reasonable and can in practice be used to estimate the BET surface area from Blaine surface area measurements.

Conclusions

From the results it would appear that one could make a reasonable estimate of a limestone related material's BET surface area from the Blaine surface area. It should be stressed, however, that such a determination cannot replace an accurate measurement of the BET surface area and should merely serve as an indication.

Acknowledgement

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