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Studies on the effect of particle size on some properties of dental stone

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Most indirect dental restorations of gold or porcelain are constructed on stone working models or dies. If these models or dies are damaged during the construction of these restorations in the laboratory, then the clinical success of the restoration will be jeopardized. This study is related to particle size, surface area, hardness and scratch resistance of twelve gypsum model or die materials. No correlation was found between particle size and hardness or scratch resistance. There was very little difference in hardness and scratch resistance between most of the materials tested.

1. Introduction

In order to construct gold or porcelain restorations for the mouth, it is necessary to make a model of the patient's teeth in the laboratory. This is achieved by taking impressions of the patient's teeth and making a model or die from the impression.

Many different materials are available to dentistry for the preparation of working models and dies [1]. A recent investigation [2] showed that gypsum-based die materials (dental stones) are used extensively by specialist dental laboratories. There are two physical forms of the hemi-hydrate of calcium sulphate, the α form (dental stone) and the β form (dental plaster) which are produced from gypsum by different manufacturing processes. The α form consists of small regular crystals, requires less water to achieve a satisfactory working consistency, and is harder.

On the addition of water, hydration occurs:

$$(CaSO_4)_2 \cdot H_2O + 3H_2O = 2CaSO_4 \cdot 2H_2O.$$

At the powder/water ratio used, a supersaturated solution of the hemi-hydrate is formed in the slurry; because the dihydrate is very much less soluble in water, crystallization occurs as is manifested by the setting of the gypsum. Dental stone fulfills most of the requirements of an ideal die material, its main problem is its poor resistance to abrasion and surface hardness [3, 4]. Some of the factors which influence hardness are the water/powder (W/P) ratio, method of mixing and the impression material into which it is poured [3]. It has been suggested that the method of manufacture and the shape of the resulting crystal could also influence the hardness of dental stone [5].

The influence of particle size on the efficiency of certain materials is well known. Coal is crushed to present a large surface area for combustion and cement is finely ground to increase the setting time. The surface detail of dental casts is improved by a reduction in the particle size of dental stone [6], whilst strength and hygroscopic expansion of dental investment material is influenced by the specific surface area, i.e. surface area per unit mass of material [7].

By varying the excess water in the mix, Mahler [8] established a definite correlation between hardness and ultimate compressive strength. Combe and Smith [5] also showed a correlation between surface hardness and compressive strength $r = 0.61$. The relationship between hardness and transverse strength was not so strong $r = 0.528$. The weakest correlation was between compressive and transverse strength, which gave $r = 0.47$. The relationship between hardness and compressive strength might be anticipated since the stresses involved are similar.

The aim of this paper is to study the average particle size and specific surface area of a variety

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TABLE I Materials tested

Material	Manufacturer or agent*
Dental Plaster	British Gypsum, UK
Kaffir "D"	Cafferata & Co Ltd UK
Alpha Dur 700 brown	*Chaperlin & Jacob Ltd, UK
Alpha Dur 700 white	*Chaperlin & Jacob Ltd, UK
Begodur	Bego, West Germany
Duralit	Degussa, West Germany
Duruston	Flexico, UK
Ferrodur	Renfert, West Germany
Glastone	Ransome & Randolph Co, USA
Super Die	Whipmix Corp., USA
Tewestone	Kettenbach, West Germany
Velmix	Kerr. Mfg. Co, USA

of dental gypsum model materials and to compare the effect of these properties on their surface hardness and scratch resistance.

2. Test methods

The materials tested and the name of their manufacturers or agents are shown in Table I. Dental Plaster of Paris was included for comparison.

2.1. Average particle size

The average particle size was measured with the Fisher Sub-Sieve Sizer. This equipment was described by Gooden and Smith [9] and was designed to measure the average particle size of a powder by what is generally known as the air permeability method. This method is based on the fact that a current of air flows more readily through a bed of coarse powder than through a bed of fine powder.

Before the average particle size of the powders was measured the true density of each gypsum material was established. The specific gravity bottle method was used with ethyl alcohol for liquid displacement. The mean of two determinations for each material was recorded. Specimens of each material were weighed in grams equal to their true density and in turn each powder was placed into the sample tube of the Fisher Sub-Sieve Sizer and packed to a constant porosity. A pilot study established that a porosity of 0.5 was suitable for all the powders tested, porosity being defined as the ratio of air space in the sample bed to the total volume occupied by the packed sample. The specimen, in the sample tube, was placed in the apparatus. With all other variables fixed the average particle size was read directly off the chart attached to the Fisher Sub-Sieve Sizer. The mean value of five determinations for each material was recorded.

2.2. Specific surface area

The specific surface area was calculated according to the formula (Fisher Sub-Sieve Sizer Catalogue no. 14-312)

$$SW = \frac{6 \times 10^4}{dmP}$$

where SW is the specific surface area in cm^3 per g material. dm the average diameter in microns taken from chart and P the true density of the material.

2.3. Preparation of specimens

Two specimens of each material were prepared from separate mixes of each material. Instructions supplied by the manufacturers were followed during the specimen preparation and the W/P ratios in Table II were used. The materials were mechan-

TABLE II The effect of average particle size on some properties of dental stones

Material	Water/ powder ratio	True density ($cm^3\ g^{-1}$)	Average particle size (μm) ($n = 5$)	Specific surface area ($cm^2\ g^{-1}$)	Scratch width (mm × 10^4) ($n = 10$)	Hardness (depth of indentation) (mm × 10^4) ($n = 10$)
Plaster of Paris	0.50	2.476	4.18 ± 0.04	5797	2776 ± 519	80.2 ± 4.0
Kaffir D	0.30	2.750	5.62 ± 0.09	3881	1262 ± 43	37.6 ± 4.3
Alphadur 700 Brown	0.24	2.606	5.24 ± 0.12	4384	1240 ± 71	46.8 ± 8.6
Alphadur 700 White	0.24	2.661	5.06 ± 0.08	4549	1293 ± 61	34.8 ± 2.4
Begodur	0.25	2.608	7.42 ± 0.11	3100	1403 ± 73	41.2 ± 4.0
Duralit	0.24	2.723	7.20 ± 0.16	3059	1315 ± 68	38.8 ± 4.1
Duruston	0.25	2.676	6.26 ± 0.28	3581	1153 ± 45	39.8 ± 7.6
Ferrodur	0.23	2.611	5.48 ± 0.08	4192	1425 ± 30	43.6 ± 5.9
Glastone	0.25	2.748	5.88 ± 0.19	3712	1232 ± 48	41.0 ± 2.5
Superdie	0.22	2.664	6.42 ± 0.29	3508	1113 ± 55	32.8 ± 4.7
Tewestone	0.24	2.664	4.90 ± 0.14	4595	1278 ± 44	44.2 ± 4.7
Velmix	0.24	2.777	5.52 ± 0.11	3990	1284 ± 32	35.0 ± 3.4

TABLE III Zero order correlation coefficients between particle size, surface area, hardness and scratch resistance

Variable	Variable			
	Particle size (Y)	Surface area (X_1)	Hardness (X_2)	Scratch (X_3)
Particle size (Y)	1	-0.972^*	-0.187	0.082
Surface area (X_1)		1	0.262	0.031
Hardness (X_2)			1	0.361
Scratch (X_3)				1

*Significant at $p < 0.01$.

ically mixed with distilled water under 68.5 cm of vacuum for 15 sec. They were poured into a polymethylmethacrylate (Perspex) mould, 50 mm x 40 mm x 5 mm which was sealed to a glass base with wax. The overfilled moulds were covered with a glass slab which was rocked into position to contact the top surface of the mould. The specimens were removed from their moulds 1 h from the start of the mix and stored in air at room temperature until a constant weight was recorded. Specimens were stored and tested at a room temperature of 23 ± 2.0° C and relative humidity of 50% ± 10%.

2.4. Scratch test

The scratch testing equipment used in this study was previously described by Harrison and Hugget, [10]. The scratch was made using a diamond tool (Taber no. 139–155) which was lapped to a 90° included angle with a 3 mm radius on the point. A 150 g load was applied to the diamond tool which was allowed to move across the specimen at a speed of 1 mm sec^{-1}. The Nikon optical microscope (Nikon, Nippon, Kogaku, Tokyo, Japan) was used to measure the width of the scratch. The same specimens were used for both the hardness and scratch tests.

2.5. Hardness

The hardness of the specimens was determined using the servo operated Wallace, Micro-Hardness Tester, model H6B/SA/C (H. W. Wallace & Co Ltd, Croydon, UK). The depth of indentation using a 3 mm diameter ball indenter under a minor and major load of 1 and 300 g, respectively, was recorded after 15 sec. Ten determinations of each material were recorded in millimeters.

Since the relationship between hardness and compressive strength of dental stones has been established previously [5, 11] it was considered unnecessary to include further tests on compressive strength in this study.

2.6. Microscopic examination

Slides were produced from powder specimens of the gypsum model and die materials, using canada balsum in xylol as a mounting medium. The prepared slides were allowed to dry completely before being examined and photographed with the Zeiss Ultaphot (Carl Zeiss (Oberkochem) Ltd, 31–6 Foley Street, London).

3. Results

The mean and standard deviations of average

TABLE IV Results of tests on dental stones [5]

Material	Wallace hardness no.	Compressive strength (10^3 psi)*	Transverse strength (10^3 psi)
Calestone	8.6 + 1.1	8.2	2.1
Kaffir D	8.1 + 0.9	10.5	3.0
Kemcal	8.1 + 0.8	9.6	2.4
Q. S. Stonehard	9.2 + 0.8	11.3	2.5
Crystacal	7.8 + 0.7	11.4	3.4
Diolite	8.3 + 0.5	9.4	2.5
Duruston	7.8 + 0.6	7.5	2.6
Glastone	7.2 + 0.8	10.5	3.1
Rockstone	7.7 + 1.0	10.0	3.9
Velmix	7.0 + 1.0	9.8	2.9

*10^3 psi = 6.89 N mm^{-2}.

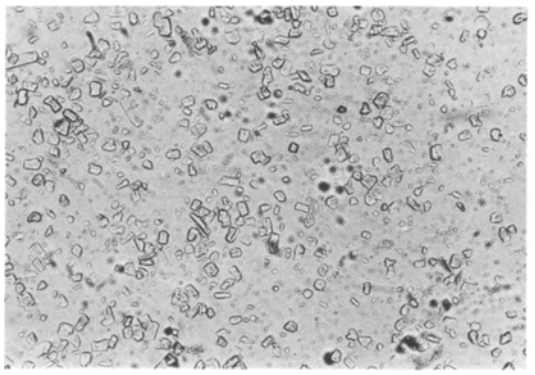

Figure 1 An example of dental stone powders, approx. × 100. This is typical of the smaller particle sizes as seen in Super Die, Kaffir "D" and Duruston.

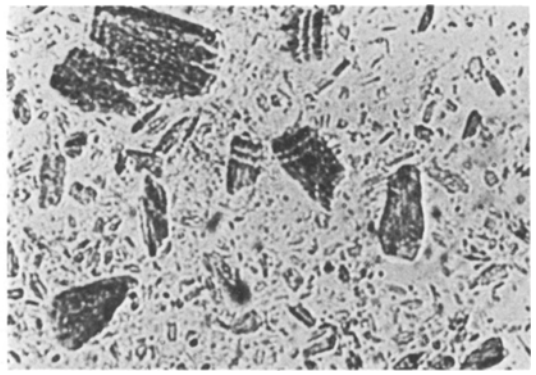

Figure 3 Plaster of Paris, approx. × 100, showing the large, irregular-shaped porous particles.

particle size, hardness and scratch resistance are shown in Table II. The harder the material the smaller the indentation and scratch figure. The table also includes water/powder ratio, true density and specific surface area. The higher specific surface area indicates the finer powder. Average particle size appeared to be inversely proportional to specific surface area for most of the results, although when plotted the figures appeared to diverge at high specific surface values. The relationship between average particle size and specific surface area, average particle size and hardness, average particle size and scratch, specific surface area and hardness, and specific surface area and scratch were examined using scattergrams and the correlation coefficients were calculated. A matrix of zero order correlation coefficients r is shown in Table III, it can be seen that a strong relationship between particle size and surface area was indicated. $r = -0.972$ which is significant at the 1% level.

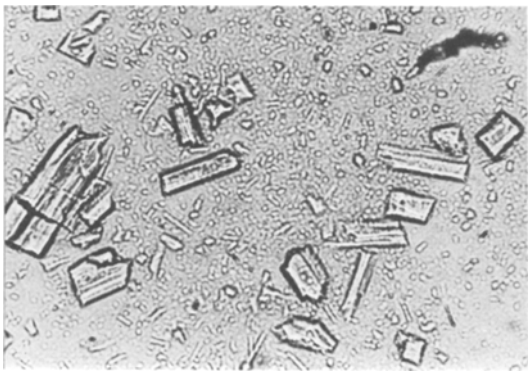

Figure 2 An example of dental stone powders, approx. × 100. This shows a typical example of the larger particle sizes as seen in Velmix, Tewestone and Glastone.

There was no evidence to support any relationship between the other variables.

The results of the hardness tests confirmed the previous work of Combe and Smith [5] (Table IV), which showed little difference between most of the stone materials.

4. Discussion

A microscopic examination of the gypsum powders showed that particle sizes varied with most of the materials. The stone materials consisted of dense, regular-shaped crystals. Superdie, Kaffir "D" and Duruston presented the smaller crystals (Fig. 1); Velmix, Tewestone and Glastone contained the larger crystals (Fig. 2). The largest crystals were noticed in Plaster of Paris (Fig. 3), these appeared irregular in shape and porous by nature.

The results (Table II), showed that Plaster of Paris recorded the smallest average particle size, therefore, the large particles viewed with the microscope must be in the minority and the bulk of the material is likely to consist of a much finer powder. Further work on particle size distribution would be necessary to confirm these results.

It is known that the rigidity of the set mass of dihydrate ($CaSO_4 \cdot 2H_2O$) is due to the development of a skeleton of interlocking crystals. The strength of the set material is dependent on the shape, size and purity of the component crystals as well as the strength of bond between the various crystals [12]. When these gypsum materials are mixed, an excess of water to that required for exact chemical combination is added to produce a workable mix. Consequently, when the dihydrate is allowed to dry out, small voids are presented

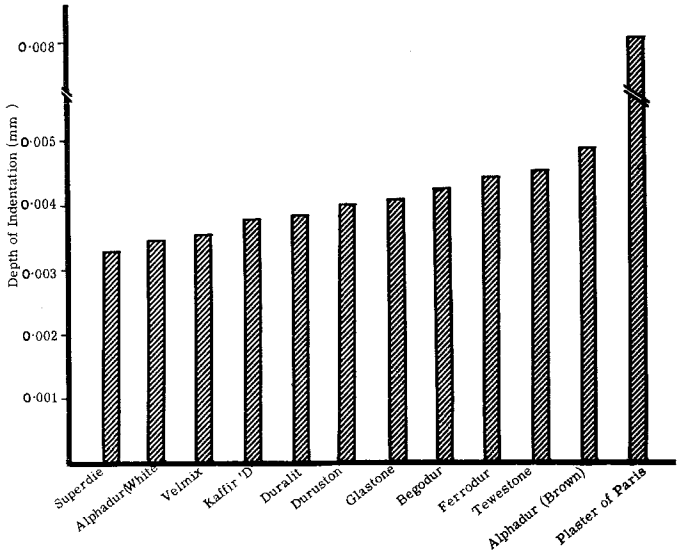

Figure 4 Hardness of dental stones.

throughout the material, thus the higher the W/P ratio the softer the material since compression allows the voids to collapse. Therefore, because plaster of paris required a high W/P ratio, it was the softest (Fig. 4) and least resistant to scratching of all the materials tested. The hardness (Fig. 4) and scratch tests (Fig. 5) showed no significant difference ($P > 0.05$) between the model stone, Kaffir "D" and the die stone Velmix. With the average particle size (Fig. 7) and true density of these two materials being so close and the W/P ratio so different, it would suggest that the method of manufacture has a big influence on the mechanical properties of these materials. The person using these materials can also influence their mechanical properties and the W/P ratio recommended by the manufacturer must be used if the maximum hardness, strength and accuracy of dental stone is to be obtained. For example, because of the small differences in die stones, a higher W/P ratio or thinner mix than that recommended by the manufacturer would cause a loss of any advantage of a slightly harder material created by better manufacturing techniques.

Various methods are available for the calculation of specific surface area from particle size distri-

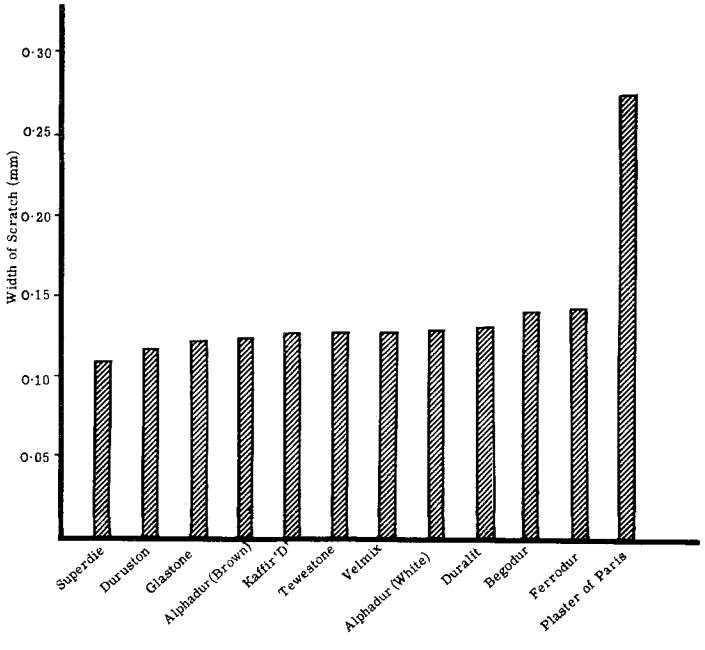

Figure 5 Scratch tests on dental stones.

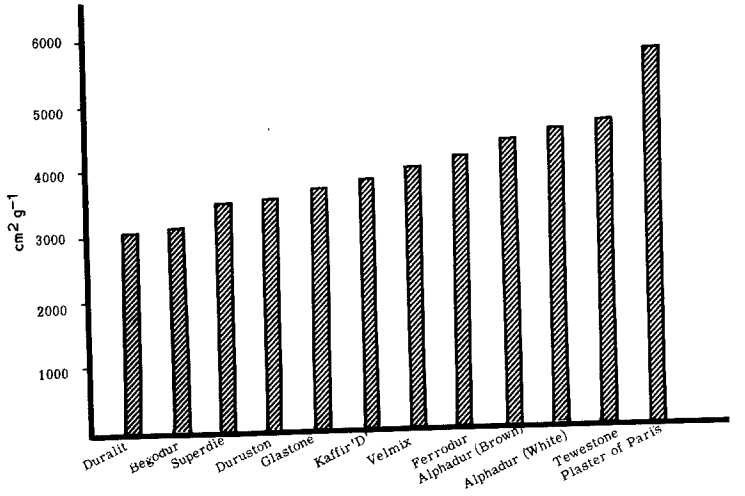

Figure 6 Specific surface area of dental stones.

bution [11]. Some methods are said to be more complex and some more accurate than others. It is considered that the accuracy of some of these methods does not justify some very elaborate procedures and a straightforward numerical method is generally adopted which can be carried out quickly with the aid of a simple desk calculator. The calculation of specific surface area from the average particle size, the method used in this study, is not generally adopted for porous materials but, because this work is essentially a comparative study of a group of similar materials, this method was considered suitable for this purpose.

It was expected that the finer powders with a low average particle size would present a large specific surface area or surface area per unit mass and the the results of thsi study confirmed this study confirmed this relationship. It can be seen that die stone materials varied in average particle size and surface area quite considerably (Figs. 6 and 7). The fact that Plaster of Paris is the softest of all gypsum model and die materials required a high W/P ratio (0.5) and presented the smallest average particle size, it would be reasonable to assume that those powders which presented the largest particle size, e.g. Begodur, should in theory

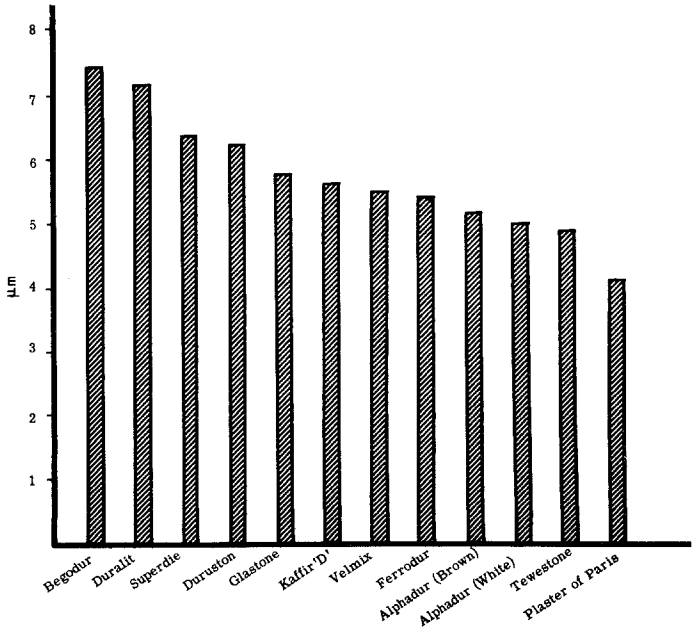

Figure 7 Average particle size of dental stones.

need less water or a low W/P ratio, to form an acceptable working mix and subsequently produce the hardest working surface.

The results of this investigation did not confirm this supposition.

5. Conclusions

(1) There was very little difference in hardness and scratch resistance between most of the Gypsum model and die materials.

(2) A strong negative relationship between average particle size and specific surface area was indicated.

(3) There was no correlation between any of the other variables.

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