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THE CAKING AND FLOW PROPERTIES OF DRY POWDERS - II

by

R. M. Forward

Summary

The crushing strength test at present used for determining the caking tendency of dry powders has been reviewed and the possible basis for a new laboratory test devised.

The effect of additives, particularly magnesium stearate, on the cohesion and water absorption properties of powders has also been investigated.

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Introduction

Early employment of sodium bicarbonate dry powders as fire extinguishing agents was of doubtful value due, in the main, to the caking properties of untreated powders. However, improved manufacturing processes developed some 15 years ago, made this medium commercially competitive with carbon dioxide and foam for controlling flammable liquid fires.

Recently the need for powders for rather different applications has been recognised. Thus, powders have been, and are still being developed:-

(1) for extinction of fires involving combustible solids as well as flammable liquids;

(2) for use in conjunction with foam. For this purpose the powder must be compatible with the foam, i.e. it must not break down the foam to any appreciable extent;

(3) for dealing with reactive metal fires.

A dry powder extinguisher should, under all normal operational storage conditions, be capable of immediate operation and the efficient discharge of its contents at a moment's notice; a suitable method of assessing the storage properties of powders is therefore essential. Part I of this note[1] describes an apparatus for measuring the discharge characteristics of dry powders from standard extinguishers. Used in conjunction with the accelerated storage cabinet, this method can give a good indication of whether the powders will remain in a readily dischargeable condition for long periods of time. The physical properties of the powders such as bulk density, moisture absorption, and cohesion have been investigated in the laboratory, and an attempt should be made to link these properties with the storage and discharge properties obtained using the apparatus mentioned above.

Properties of powders

The method at present in use for determining the caking tendency of dry powder extinguishing agents consists of determining the force necessary to break up a pellet of powder which has been conditioned by exposure to atmospheres of varying humidity.

A pellet 2.54 cm in diameter and 1.5 cm high, giving a volume of 7.6 cm^3 is prepared by compressing the powder in a cylinder fitted with a plunger. The pellet is placed in atmospheres of 92% relative humidity and 0% relative humidity for 6 and 24 hours respectively, and its crushing strength then measured by gradually increasing the force on a plunger of area 1 cm^2, placed in the centre of the top surface, until the pellet collapses.

One of the difficulties involved in this method is deciding upon the value of the packed density to be used in making the pellet. The standard test consists of allowing a 50 gm sample, contained in calibrated cylinder, to fall 40 times through a height of 4 cm on to a metallic base. From the volume and mass obtained, the density used in making the pellet is determined.

The dependence of the crushing strength on this density can be seen from Figure 1 in which a standard commercial powder was used and the force necessary to collapse the untreated pellet measured for values of the bulk density ranging

from 1.250 to 1.525 gm/cm^3. A similar curve is shown on the same graph for pellets, treated by placing in turn in humidities of 92% and 0% relative humidity, for 6 and 24 hours respectively, as specified in the test method. The dependence of the crushing strength on the bulk density is seen to be the same for treated and untreated pellets.

In the case of some powders, tamping the sample 40 times is sufficient to reduce it to very nearly its minimum volume. This, however, is not true in all cases, as can be seen from Figure 2. It is safe to assume that the maximum bulk density will be reached when the powder is stored in an extinguisher for a long period of time and, therefore, this figure rather than the density at 40 taps should be used in any determination of the cohesive forces preventing the disruption of the powder.

The packed density is dependent on three properties of the powder

(1) the particle shape,

(2) the particle size,

(3) the water-proofing or bulking additives,

used to improve the flow and moisture repellant properties. The powders used for fire extinguishing agents generally have irregular shaped particles and the bulk density is, therefore, considerably less than if they were spherical. For spherical particles, it may be shown that, for a close packing arrangement, the bulk density would be 74% of the solid density. The maximum density obtained for any of the powders measured, namely for potassium sulphate, was 67% of the solid density; for sodium bicarbonate-based powders of the same surface area, the maximum figure was 63%, indicating that the Na HCO_3 has less regularly shaped particles.

Addition of a small percentage of stearate or other similar long chain fatty acid will give a marked increase in the bulk density. Thonzeau and Taylor(2) show an increase of between 15 and 20% on dredged samples of stone dusts used for suppressing explosions in mines.

An increase in surface area or decrease in particle size results in an increase in bulk density in agreement also with the results of Thonzean and Taylor, who determined a correlation coefficient between the two properties, intending to use the bulk density as a ready measure of the specific surface area of any individual dust.

A relation similar to the one derived by these workers for limestone dusts has been obtained for a sodium chloride-based powder:-

$$S = 1.83 - 1.38 \Delta$$

where S = specific surface area (m^2/gm) and

Δ = bulk density gm/cm^3.

This equation holds for values of the surface area likely to be encountered in fire-fighting powders, i.e. between 0.1 and 0.4 cm^2/gm.

As an alternative to the tamping method of determining bulk density, a vibration technique was tried. A graduated cylinder containing a weighed mass of powder was mounted on the end plate of an electro mechanical vibrator and the sample vibrated at 50 cycles/sec with an amplitude varying from 0.001 to 0.02 in. The bulk density was found to increase up to a value of the amplitude of approximately 0.01 in, after

which the excessive vibration tended to disrupt the bed.

This vibration technique was used in another test for measuring the cohesive forces in a bed of powder, at present in its early stages of development. A diagram of the apparatus is shown in Figure 3. Fifty grms of powder is introduced into the perspex cell and mounted on the vibrator - amplitude 0.005 in frequency 50 cs/sec - for 5 minutes or sufficient time for it to reach its minimum volume. The plug in the base is then carefully removed and the pressure in the body increased until a hole is blown through the bed. The pressure necessary to disrupt the bed is read off on a monometer. This gives a measure of the cohesive forces holding the particles of powder together. Figure 4 shows the effect of increasing the quantity of stearate added to a bicarbonate based powder on these cohesive forces. The continuous line is the plot of the results obtained with the above method, including 95% confidence limits, while the dotted line shows the same effect measured using the crushing strength method.

No complete data are yet available for the caking effect due to moisture using this method, but preliminary results show that, for a good free-flowing water-proofed powder, humidification treatment results in a twofold increase in the pressure necessary to disrupt the bed, while, for a poor powder with low stearate concentration, the increase is tenfold or greater. This method eliminates the difficulties of deciding on a value of the bulk density to be used in preparing the bed, and also the inconsistencies introduced through the depth of the bed when using a plunger.

Apart from the cohesion of particles within the bed due to Van der Waal's and interlocking forces, adsorption of moisture has a very significant effect on the ease with which a sample of powder may be fluidised. Forces of attraction between polar water molecules adsorbed on to the surface of the powder increases the stickiness to a marked extent. Stearate, coated on to the surface, as well as decreasing the friction between the particles and so making the bed more fluid, also prevents the adsorption of water on to the surface due to the non-polar nature of the hydrocarbon chain. The effectiveness of the treatment with stearate is related to the efficiency with which the manufacturer applies it and also to the moisture adsorbing property of the chemical employed.

If the additive is simply stirred in with the base chemical, little or no protection from moisture is afforded to the powder. The additive, to be effective, has to be ground on to the surface and spread evenly over the particles of powder.

Assuming a value of 0.3 m^2/gm for the surface area of the powder to be water-proofed, and a value of 20.5(3) square Angstrom units for the area of 1 molecule of fatty acid, the amount of stearate necessary to coat the powder with a monomolecular layer can be shown to be 0.074 percent.

From Figure 5 it can be seen that 0.1 percent stearate has a considerable effect on the proofing of sodium bicarbonate powder.

Since moisture has such a large effect on the cohesive nature or "stickiness" of the powder, an apparatus, pictured in Plate 1, was made up in which the increase in moisture can be observed continuously with the powder sample contained in an atmosphere of constant temperature and humidity. The temperature is controlled thermostatically and is maintained at the desired level by means of a reservoir heated by a 100 watt electric mantle. The humidity is maintained at any required level by saturated solutions of inorganic salts placed in the base of the cabinet.

The powder sample, in the form of a pellet of approximately 2.5 gm weight, is suspended from a calibrated silica spring which obeys Hooke's Law up to a load of 5 gm. The sensitivity is approximately 2 cm/gm and, with the aid of a

travelling microscope, the increase in weight can be determined to the nearest 0.5 mgm.

The effect of stearate on the water absorption properties of powders is illustrated in Figure 5 in which the increase in weight of pre-dried sodium bicarbonate pellets, treated with stearate from 0.0 to 0.5 per cent in an atmosphere of 92 per cent relative humidity at 25°C, is plotted against time. It can be seen that any increase above 0.1 per cent has little effect on the water-proofing and all samples come to equilibrium with the atmosphere after about 30 hours exposure. Figure 6 shows the change in crushing strength for the same samples of stearated bicarbonate, with increase in the period of humidification. This crushing strength is a measure of the combined effects of improved fluidity, and the improved moisture repellant properties, both due to the stearate.

Preliminary experiments showed that the rate at which moisture was adsorbed was practically independent of the bulk density for values suitable for making up coherent pellets, but increased with decrease in particle size. This is evidently due to the increase in area available for absorption.

D. J. Rasbash(4) has shown that below 80 per cent R.H. only minor caking effects were apparent in samples of limestone dust admixed with various percentages of sodium bicarbonate. This was due to the low moisture adsorption at humidities below this value. Eisner, Fogg and Taylor(5) found that the cohesion of limestone powders, measured by a tilting plate method, increased rapidly at a value of 84 per cent R.H., at which value it was assumed that the whole of the surface of the particles was covered with a moisture film. The humidity at which condensation takes place is probably related to the humidity existing over a saturated solution of the salt in question. Thus, an investigation of the water proofing of sodium chloride powders showed that, at humidities over 75 per cent, condensation of globules of water occurred on the surface of the pellets, while at 72 per cent R.H. virtually no increase in weight was observed.

It is evident that, in any determination of the caking tendency of powders, the rate at which moisture is absorbed, and the time to reach an equilibrium value with high humidity atmospheres, should be taken into account.

Conclusion

It has been shown that the method of measuring the bulk density used in the determination of the crushing strength needs modification, the maximum equilibrium density being a better measure than the density at 40 taps. The time of humidification similarly needs revision to take into account the rate at which different powders reach equilibrium with high humidity atmospheres. An alternative to the method of measuring crushing strength, which obviates the problem of determining bulk density, has been described but needs further development.

These cohesive forces and the effect of moisture adsorption should be correlated with extinguisher discharge and storage tests described in Part I of this note.

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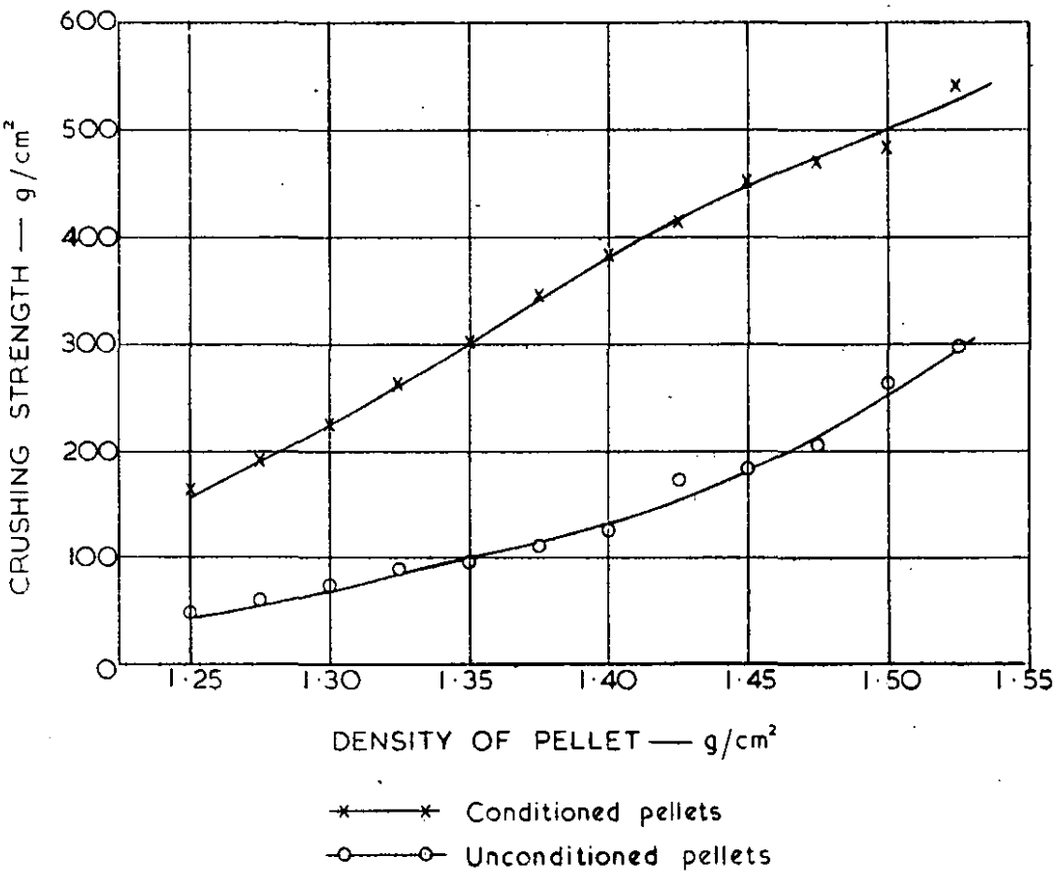

FIG. 1. VARIATION OF CRUSHING STRENGTH WITH DENSITY OF PELLET

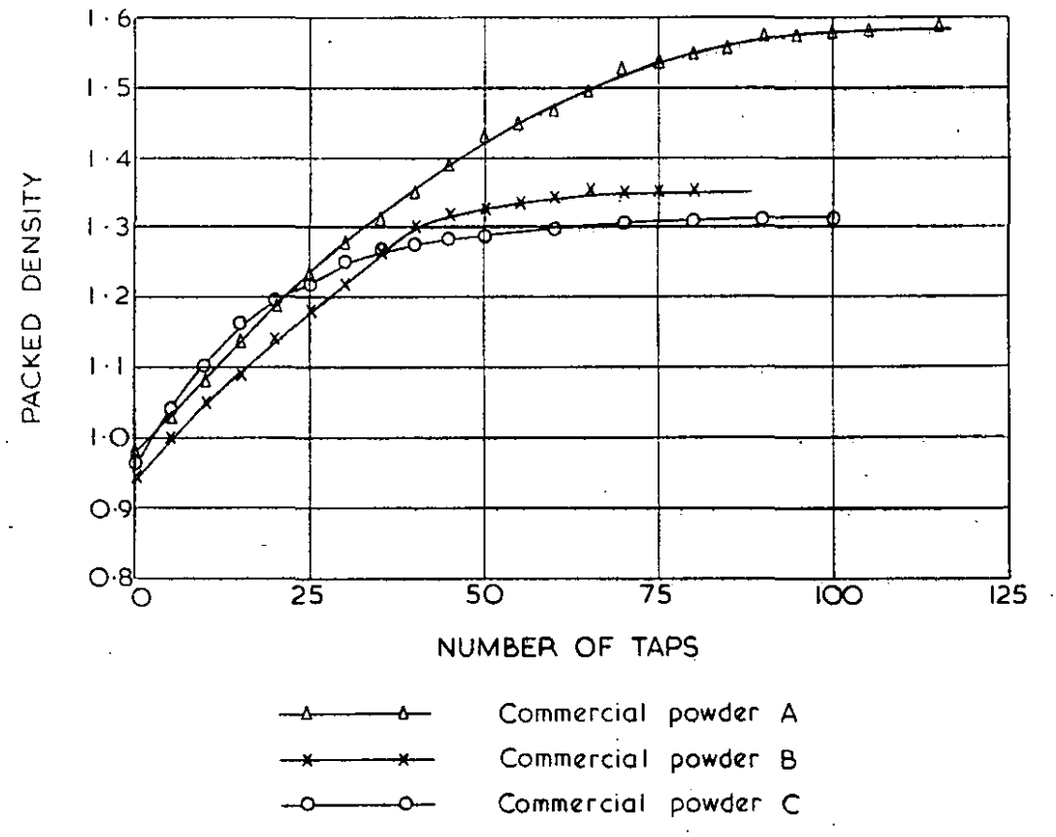

FIG. 2. VARIATION OF PACKED DENSITY WITH TAMPING

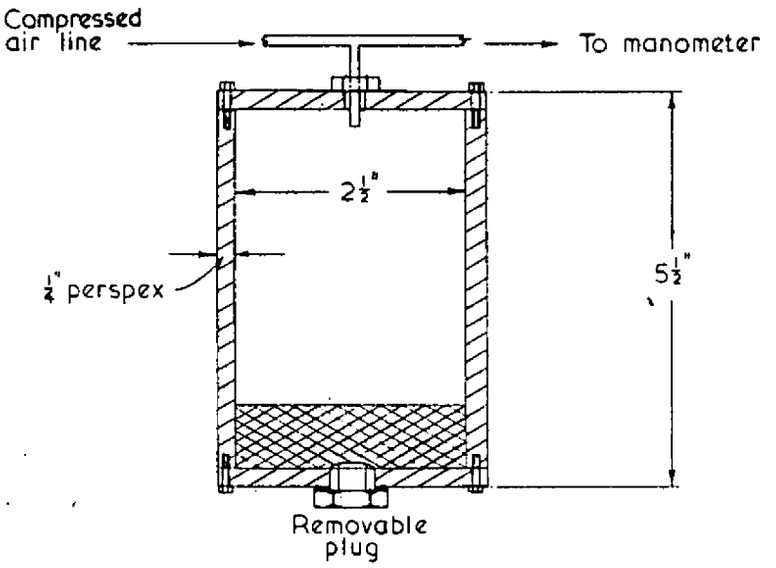

FIG. 3. APPARATUS FOR DETERMINING COHESIVE FORCES WITHIN A BED OF POWDER

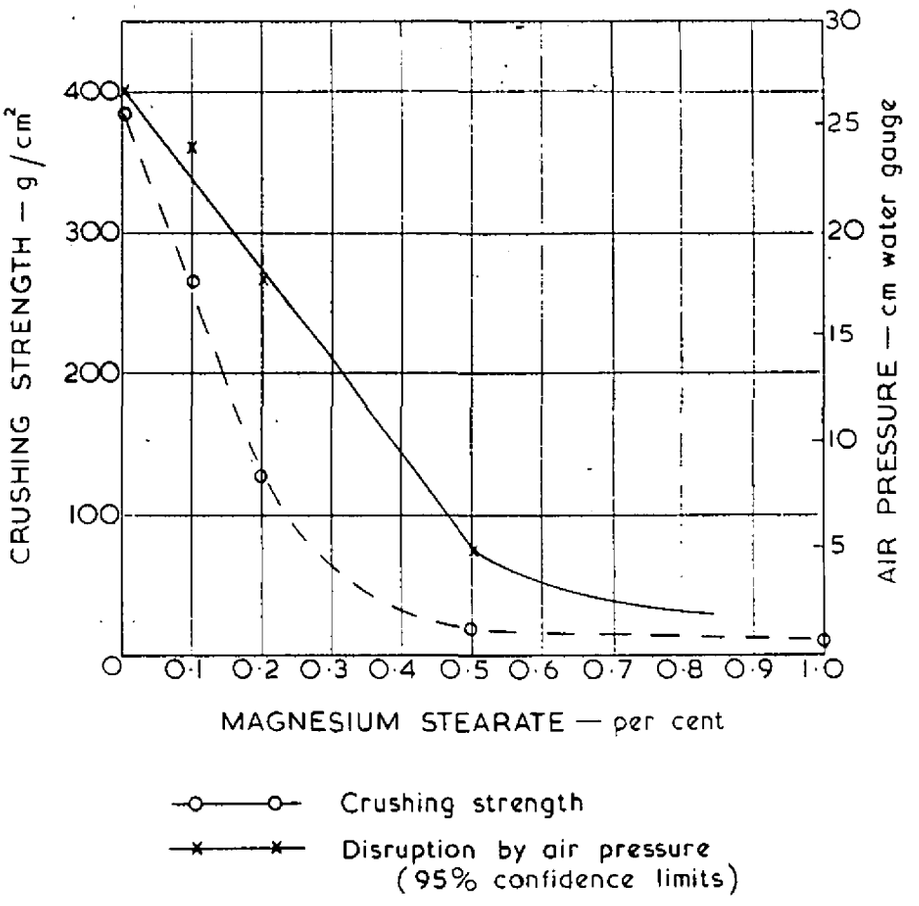

FIG. 4. EFFECT OF STEARATE ON COHESION OF SODIUM BICARBONATE POWDER

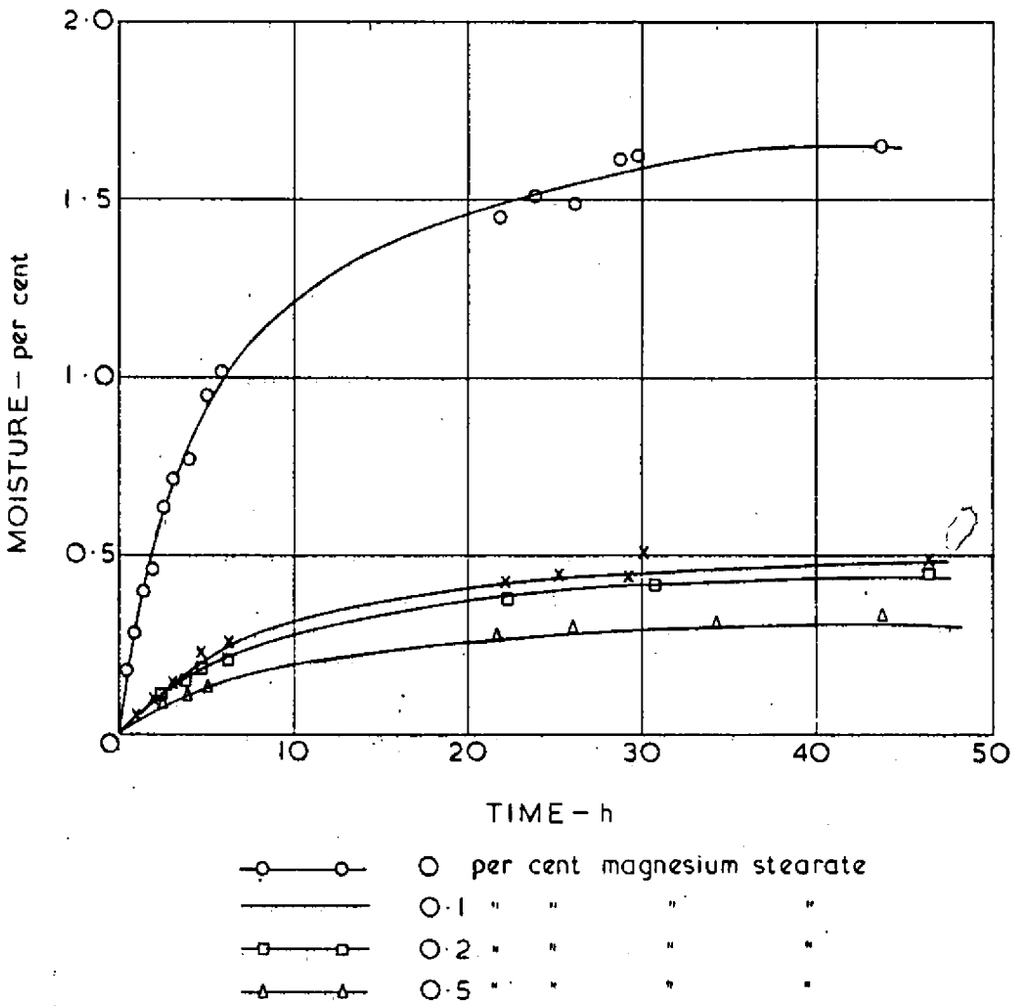

FIG. 5. MOISTURE ADSORPTION OF STEARATED SODIUM BICARBONATE

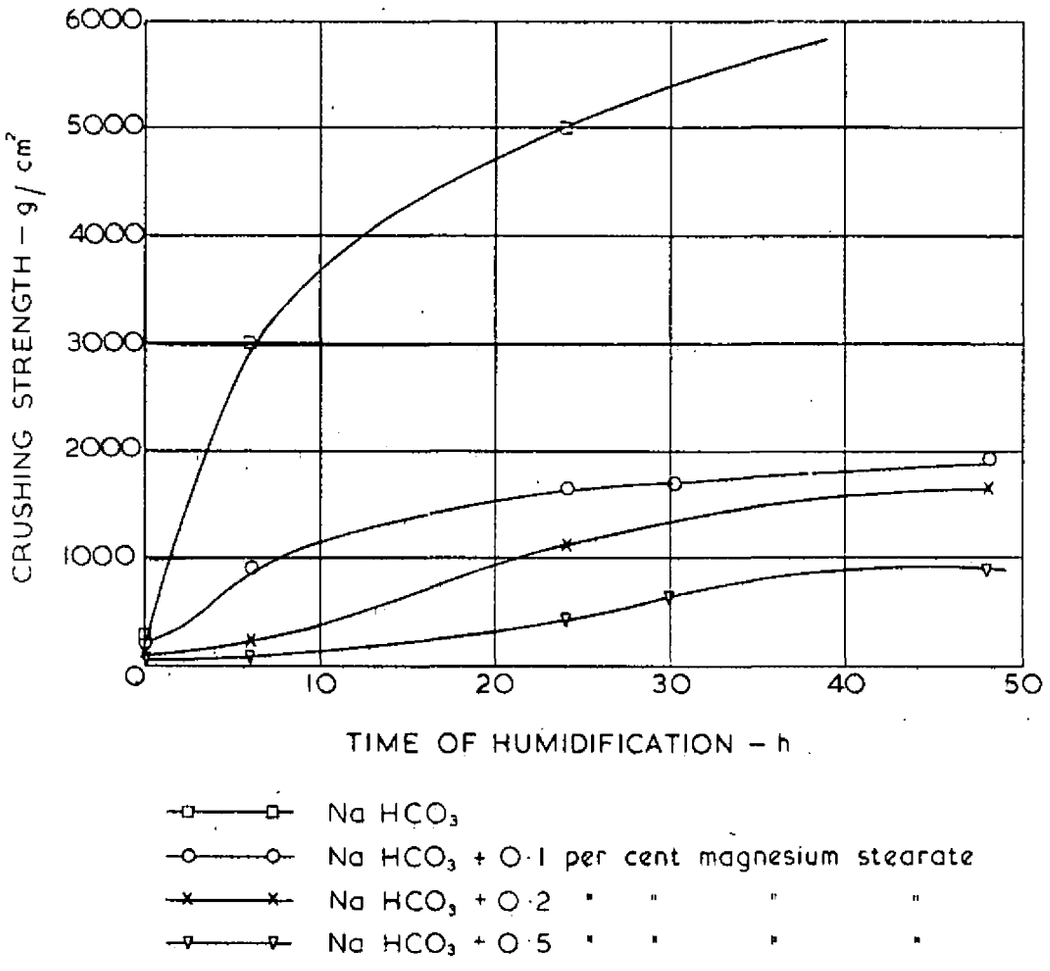

FIG.6. EFFECT OF TIME OF HUMIDIFICATION ON CRUSHING STRENGTH

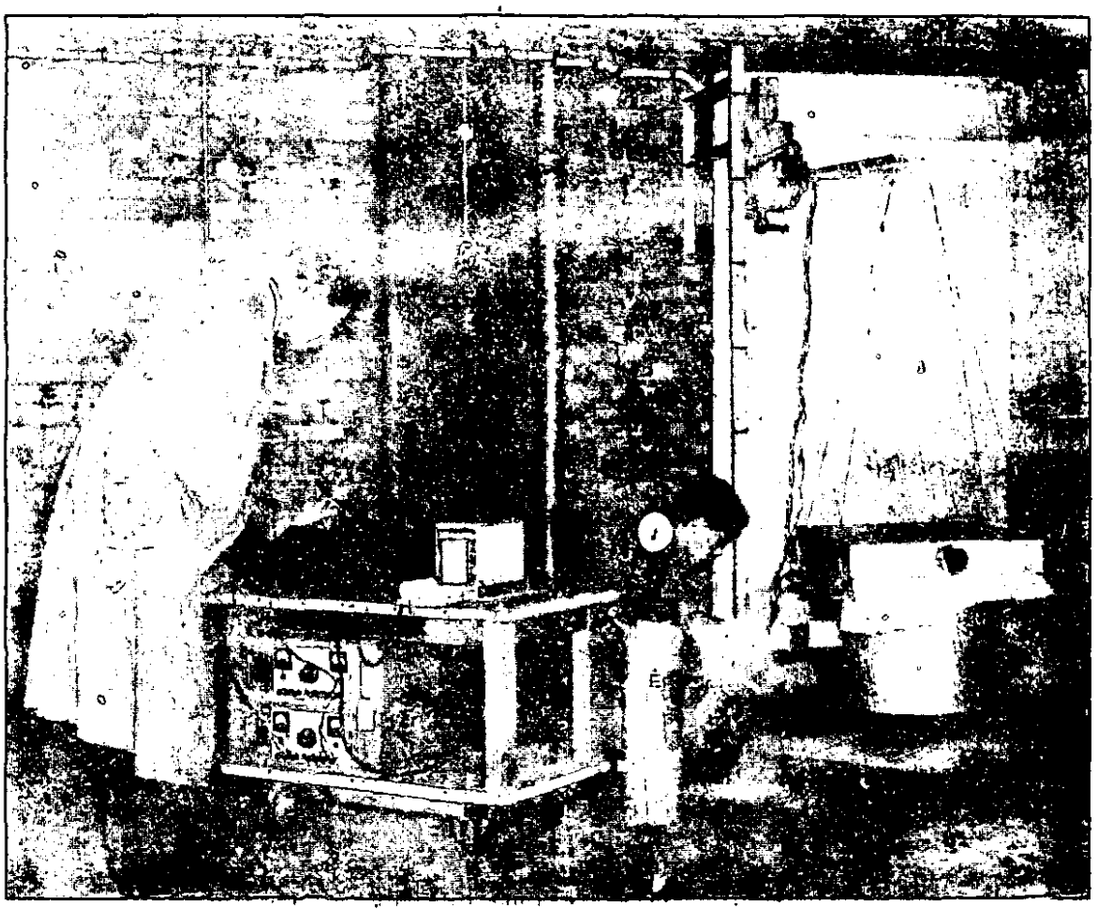

PLATE 1. CONTINUOUS MEASUREMENT OF RATE OF DISCHARGE OF POWDER EXTINGUISHER.