

# The Chemistry and Technology of **GYPSUM**

**R. A. Kuntze**, editor

**ASTM** **STP 861**

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# THE CHEMISTRY AND TECHNOLOGY OF GYPSUM

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Atlanta, GA, 14–15 April 1983

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## Foreword

The symposium on The Chemistry and Technology of Gypsum and Gypsum Products was presented at Atlanta, GA, 14–15 April 1983. The symposium was sponsored by ASTM Committee C-11 on Gypsum and Related Building Materials and Systems. Richard A. Kuntze, Ontario Research Foundation, presided as chairman of the symposium and is editor of the publication.

## **Related ASTM Publications**

**Masonry: Materials, Properties, and Performance, STP 778 (1982), 04-778000-07**

**Extending Aggregate Resources, STP 774 (1982), 04-774000-08**

**Cement Standards—Evolution and Trends, STP 663 (1979), 04-663000-07**

**Significance of Tests and Properties of Concrete and Concrete-Making Materials, STP 169B (1978), 04-169020-07**

## A Note of Appreciation to Reviewers

The quality of the papers that appear in this publication reflects not only the obvious efforts of the authors but also the unheralded, though essential, work of the reviewers. On behalf of ASTM we acknowledge with appreciation their dedication to high professional standards and their sacrifice of time and effort.

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## The Relationship Between Water Demand and Particle Size Distribution of Stucco

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**REFERENCE:** Luckevich, L. M. and Kuntze, R. A., "The Relationship Between Water Demand and Particle Size Distribution of Stucco," *The Chemistry and Technology of Gypsum, ASTM STP 861*, R. A. Kuntze, Ed., American Society for Testing and Materials, 1984, pp. 84–96.

**ABSTRACT:** In gypsum board production, the amount of water required for a stucco slurry with a suitable fluidity is considerably greater than the amount necessary for hydration of the stucco. Since excess water must be removed by drying, it is advantageous, from an energy point of view, to reduce the consistency or water demand of stucco. Constant board density is maintained thereby through increased addition of foam.

The high water demand of stucco produced by conventional calcination methods is due largely to the ability of the stucco to disintegrate, a process during which larger stucco particles break down into smaller particles upon contact and subsequent mixing with water. The water demand of stucco may be reduced by aging, that is, the exposure of stucco to water vapor over a period of time, which reduces the ability of the stucco to disintegrate. However, aging also has a negative effect on both setting characteristics of the stucco and, as a result, on the strength of the gypsum formed.

The objective of the work described in this paper was to study the relationships between water demand and particle size distribution as affected by changes in disintegration during aging. Particle size analysis and several methods of consistency measurement were used for this purpose. It was found that a direct relationship exists between water demand of stucco and particle size distribution as defined by percent disintegration, with minor deviations at initial stages of aging. In contrast, nonlinear relationships were found, with preliminary experiments, for stuccos containing aridizing agents or surfactants, which indicates that the water demand of stucco is also affected by surface properties. The water demand of stucco is not a function of the surface area of the stucco, that is, other properties, such as particle shape and surface characteristics, play a significant role. Similarly, the magnitude of the differences in water demand of various types of stucco indicates that particle size distribution, although the major factor, is not the only one influencing this property.

**KEY WORDS:** gypsum, stucco, disintegration, water demand, consistency

Modern gypsum board production is a complex process that involves the preparation of an aqueous slurry by mixing calcined gypsum (stucco) with water

<sup>1</sup> Associate research scientist and director, special programs, respectively, Ontario Research Foundation, Sheridan Park, Mississauga, Ontario, Canada L5K 1B3.

and a variety of additives such as foam, starch, surfactants, fibers, and set control agents. The slurry is cast between two layers of paper, and the board is formed by molding both paper and slurry into the desired shape and dimension and allowing the stucco to harden by hydration. The formed board consists of a wet gypsum core, lined front and back, and at both edges by paper. The core at this stage contains excess water because more water is required for slurry preparation than is necessary for hydration of the stucco. This excess water must be removed by drying through the paper, an energy intensive process. From the point of view of energy conservation, it is obvious that it would be advantageous to reduce the consistency or water demand of stucco, that is, the amount of water that must be added to the stucco to produce a slurry that can be poured. The amount of water reduced would be replaced by increased addition of foam to maintain the required core density and board weight.

Methods of reducing the water demand of stucco exist, including aging, that is, exposure of the stucco to water vapor over a period of time [1,2], spraying liquid water into the stucco immediately after calcination [3,4], introducing deliquescent salts into the calcination kettle [5], and the use of surface active agents. However, these methods generally have deleterious effects on other aspects of the board-making process. For instance, an aged stucco yields a gypsum with a lower strength than gypsum obtained from fresh stucco, and aridizing salts may cause deterioration of the bond between board core and paper.

A fresh stucco, in addition to having a high water demand also has the characteristics of high disintegration, that is, upon contact with water, large stucco particles are broken down into smaller particles [6,7]. The degree of disintegration that a stucco undergoes is increased by mechanical mixing. Also, the ability of the stucco to disintegrate is progressively lost with aging. Thus, aging affects both water demand and disintegration in a similar fashion, that is, both decrease. For this reason, it has been suggested that a relationship exists between the two [8].

The nature of this relationship is the focus of this paper. Particle size distribution data were used to determine disintegration as a function of mechanical mixing and of aging. Similarly, several methods of consistency measurement, employing various types of mixing, were used to determine water demand as a function of aging.

## Experiments

A relatively pure form of natural gypsum (Terra Alba) consisting of 98.6%  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  was used for this study. The gypsum was ground to pass a 150- $\mu\text{m}$  (No. 100) sieve and calcined in a laboratory kettle designed for both continuous and batch operation. In this manner, three 15-kg lots of stucco were prepared by continuous calcination, and three 5-kg lots of stucco were calcined by the batch process. To two lots of the latter, either calcium chloride or surfactants were added.

TABLE 1—Time required to reach limiting values (average of three calcinations).

Consistency Measurement	Time Required for Consistency to Reach Limiting Value, approximate days	Time Required for Disintegration to Reach Limiting Value, approximate days
DIN	6	4
Pouring	15	10
7 s	10	10
60 s	21	15

After calcination, the stuccos were cooled to room temperature with constant stirring to remove all entrained water vapor. The residual gypsum content and soluble anhydrite content as determined by phase analysis [9] were always less than 4.0 and 2.0%, respectively.

The stuccos prepared in the laboratory were aged at 21°C and 65% relative humidity for various periods of time. Two stucco properties, that is, consistency and disintegration, were determined immediately after calcining and after various periods of aging. These tests were continued until equilibrium values had been reached; the times required to reach these values are given in Table 1. The consistency test methods and the techniques to obtain particle size data, as well as the treatment of these data, are described briefly below.

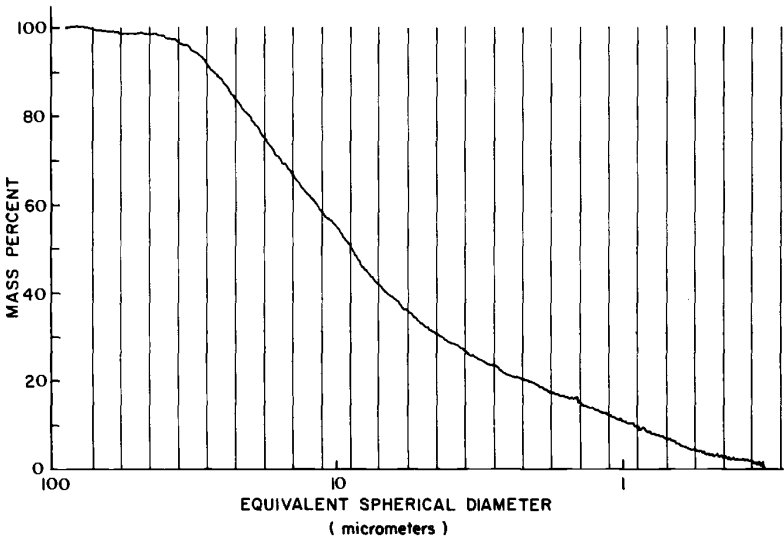
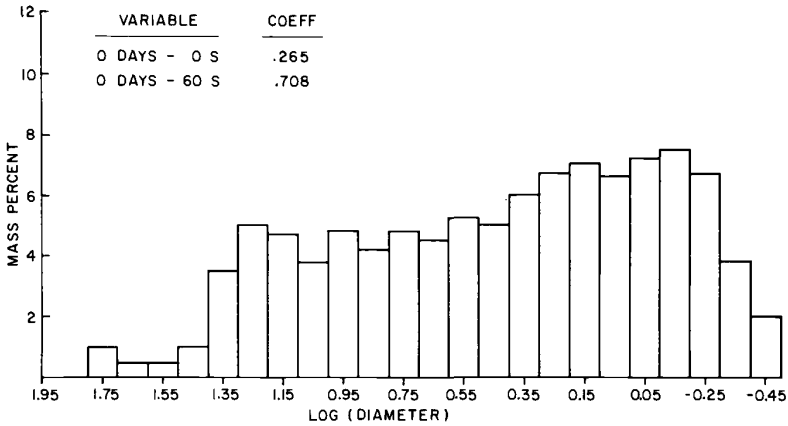


FIG. 1—Particle size distribution curve.



### Consistency Procedures

All consistency measurements were conducted by adding 0.2-g sodium citrate to 100 mL of gaging water to prevent premature stiffening caused by hydration of the stucco.

#### DIN Consistency

This type of measurement is widely used in Europe [West German Industrial Standard (DIN) Test Methods for Gypsum Plaster (DIN 1168)] to determine the so-called water:stucco factor, that is, the amount of stucco in grams that can be wetted by 100 mL water. The method consists of sprinkling stucco into the water at a specific rate without mechanical mixing. Therefore, it differs from other consistency measurements in that only spontaneous disintegration occurs.

#### Pouring Consistency

It is common practice in North America to carry out consistency measurements based on the fluidity of water:stucco slurries. The method used for the present work consisted of adding a specified amount of stucco to water and mixing both by hand for 30 s after a brief soaking period. The resulting slurry is poured onto a glass plate to form a patty. The water:solid ratio producing a patty with a specified dimension is taken as the pouring consistency. In this measurement, both spontaneous disintegration and a minimal amount of induced disintegration occur.

#### 7-s Consistency

This consistency measurement is also commonly used in North America. Stucco:water slurries are mixed for 7 s in a high-shear Waring® Blendor to simulate

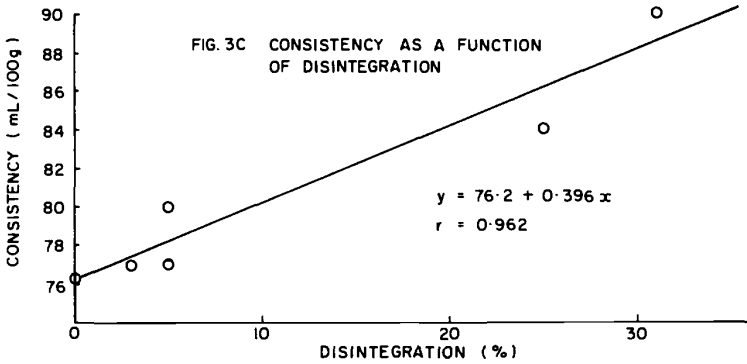
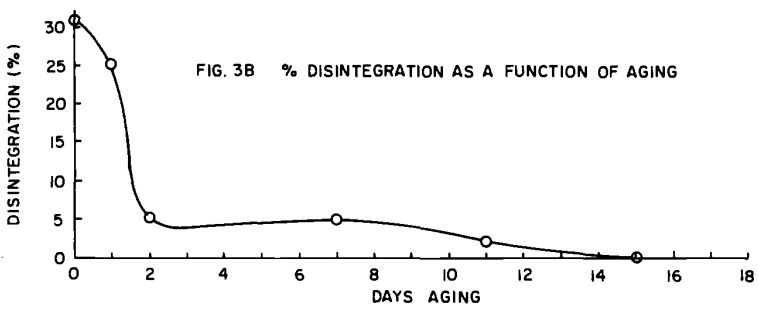
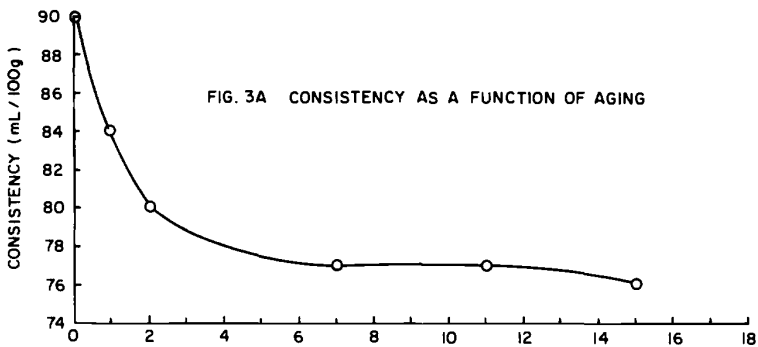


FIG. 3—Terra Alba DIN consistency.

the mixing that occurs in a pin mixer during gypsum board production. The correct consistency is expressed as the water:solid ratio that produces a slurry with a specified penetration as determined by the modified Vicat apparatus [apparatus used in ASTM Physical Testing of Gypsum Plasters and Gypsum Concrete (C 472)]. Because of the high mechanical energy involved, a significant amount of induced disintegration is obtained in addition to spontaneous disintegration.

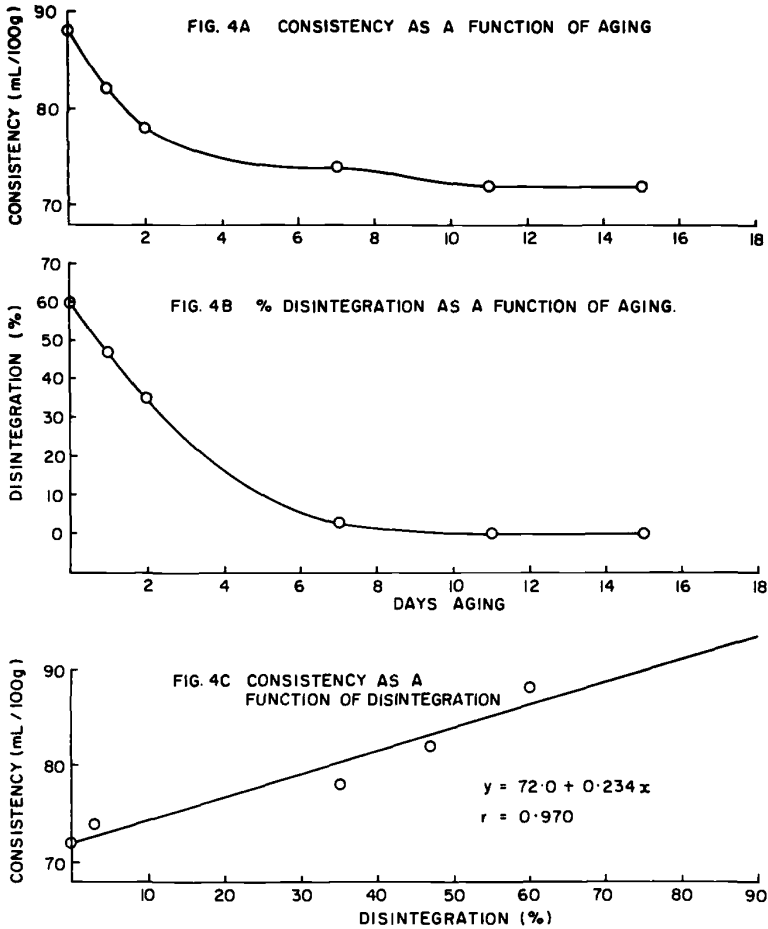


FIG. 4—Terra Alba pouring consistency.

#### 60-s Consistency

The mixing procedure used in this test, that is, 60 s in a high-shear Waring Blendor, is intended to produce maximum disintegration of the stucco particles [6]. The viscosity of a slurry mixed by this method if measured by a Ford® cup viscometer, and the water: solid ratio with which a specified flow time is obtained is taken as the consistency.

#### Disintegration Measurements

To determine the degree of disintegration occurring during the consistency procedure, the stucco slurry was mixed immediately after the test with at least

six times its volume of methanol. After mixing, the stucco solids were filtered, washed with acetone, dried, and stored in a sealed container. Control specimens, that is, stucco not subjected to disintegration, were also collected after each calcination and each aging period and stored after drying in sealed containers before particle size analysis.

#### *Particle Size Analysis*

The particle size distribution of the dry stucco was measured using a Sedi-graph® 5000D particle size analyzer. Specimens were prepared by dispersing the stucco in Sedisperse® A-11 and placing the suspension in an ultrasonic bath for 2 min. Data were acquired in the form of a cumulative mass percent versus equivalent spherical diameter curve. As demonstrated in Fig. 1, this analysis included particles with an equivalent spherical diameter as small as 0.325  $\mu\text{m}$ . For all specimens tested, the particles had equivalent spherical diameters smaller than 90  $\mu\text{m}$ .

#### *Particle Size Data Treatment*

*Determination of Percent Disintegration*—For unaged stucco that had not been subjected to the disintegration procedure, the majority of the particles were in the larger size range (5 to 70  $\mu\text{m}$ ). After disintegration to the greatest possible degree, that is, without aging and with 60 s mixing in the Waring Blendor, the majority of the particles were in the smaller size range (0 to 5  $\mu\text{m}$ ). Both were taken as having a particle size distribution representative of 0% disintegration or 100% disintegration, respectively. Specimens that had been subjected to aging or less than total disintegration or both had a particle size distribution in between these two limits, representing a specific percentage of disintegration. To calculate this percentage, a regression analysis was performed. For example, Fig. 2 represents a histogram for a specimen that had not been aged but had been mixed for 7 s. The regression analysis shows the degree of disintegration of this specimen as 72.8%. This method of regression analysis was performed on all specimens whereby the correlation coefficients obtained ranged from 0.7 to 0.9.

### **Results and Discussion**

Figures 3 through 6 illustrate the effect of aging time on consistency and disintegration, as well as the relationship between consistency and disintegration for continuously calcined Terra Alba. These data show that both consistency and disintegration decrease with time and suggest a linear relationship exists between these properties. Regression analysis of the data yields slopes, intercepts, and correlation coefficients of these curves as given in Table 2. Similar data for batch calcined stucco are summarized in Table 3.

There is a slight deviation from a linear behavior at early aging periods. For instance, Fig. 7 shows the particle size distribution for continuously calcined Terra Alba dispersed for 7 s at zero- versus one-day aging. The particle size



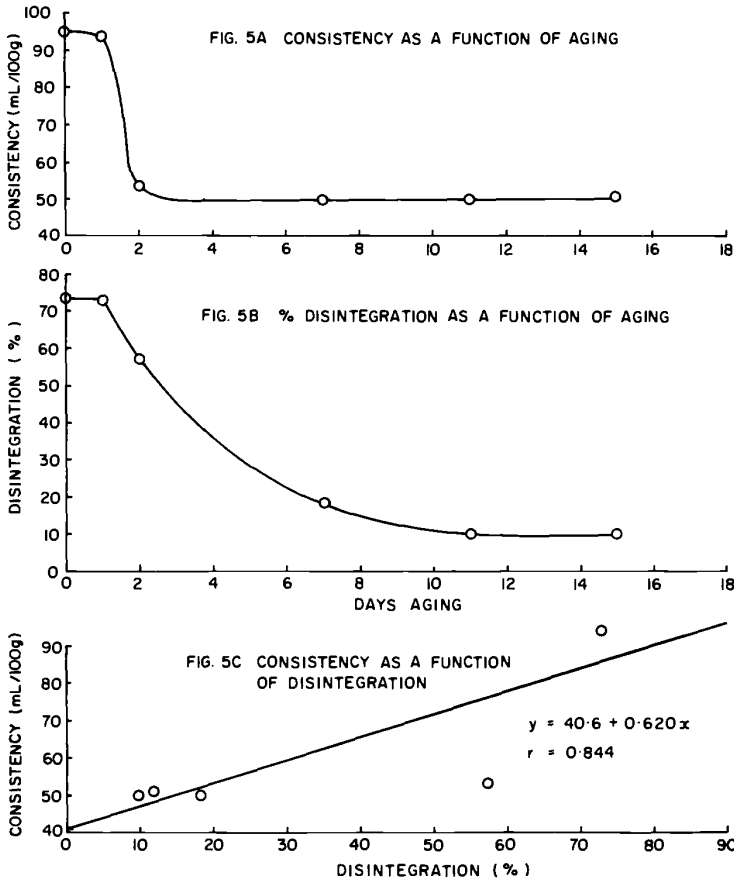


FIG. 5—Terra Alba 7-s consistency.

distribution at one-day aging shows a higher mass percentage in the 1 to 2  $\mu\text{m}$  range and a lower mass percentage in the 0 to 1  $\mu\text{m}$  range. Thus, even though one-day aging decreases the mass percentage of submicrometre particles, the stucco still disintegrates to a large degree. Therefore, at low periods of aging, almost complete disintegration may be achieved simultaneously with a lowered consistency.

The intercept is an indication of slurry viscosity. The 60-s consistency slurries are most fluid and consequently have the greatest intercept. The DIN and pouring consistency slurries are of comparable fluidity, and both are less viscous than 7-s consistency slurries. The intercept of the latter has the lowest value.

The fact that the consistency is a direct function of the disintegration suggests that at a given fluidity, the thickness of the water layer around each particle is

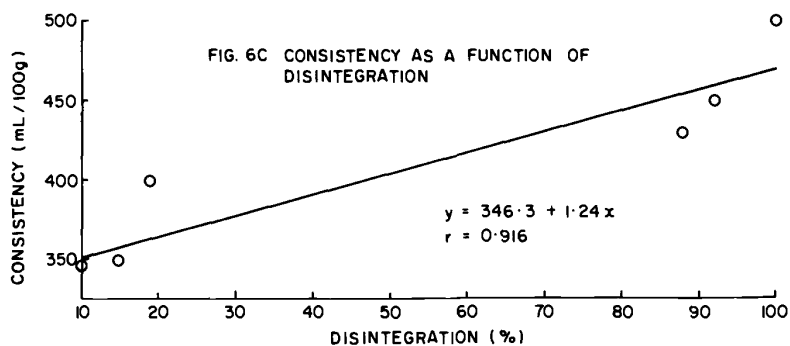
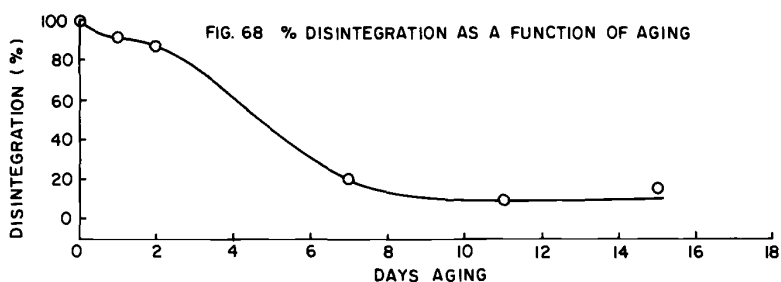
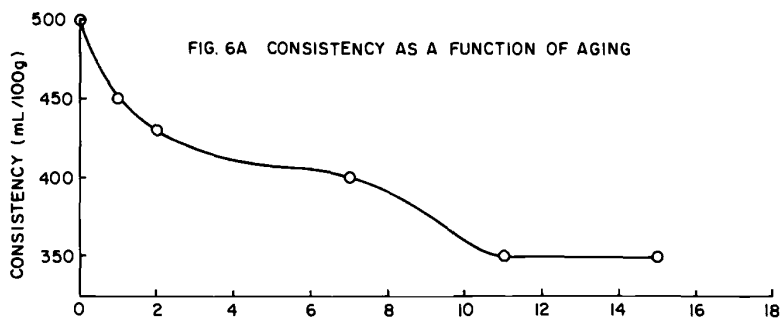


FIG. 6—Terra Alba 60-s consistency.

constant, that is, the volume of water required to achieve a certain fluidity is proportional to the surface area of the particles. If this is true, then the thickness of the water layer around each particle can be estimated by dividing the volume of water consumed by the total surface area of the particles. However, as shown in Tables 4 and 5, the thicknesses of the water layer are not constant but range is from 3.9 to 24.7  $\mu\text{m}$  for completely disintegrated stucco and from 9.2 to 90.8  $\mu\text{m}$  for undisintegrated stucco at various fluidities. The water layer thickness in slurries of continuous calcined stucco is smaller than that of batch calcined material.

TABLE 2—*Terra Alba continuously calcined consistency/disintegration regression data.*

Consistency Measurement	Slope, mL/100 g	Intercept, mL/100 g	Correlation
DIN	0.396	76.2	0.962
Pouring	0.234	72.0	0.970
7 s	0.620	40.6	0.844
60 s	1.24	346.3	0.916

The magnitude of the water layer corresponds to between 700 and 15 000 water molecules and suggests that in all slurries the particles are widely separated by a water layer of the same thickness as the particles themselves, thus isolating the particles from each other.

The fact that the water thickness is not constant may be partially explained by changes in particle size, size distribution, and shape. For example, a decrease in particle size upon disintegration may result in closer packing of the particles with a corresponding reduction of the water thickness required around each particle to yield a given slurry fluidity. Similarly, if undisintegrated particles have an irregular shape, their surface area will be greater than that of spherical particles, requiring a thicker water layer to maintain the same slurry fluidity.

The addition of calcium chloride or surfactants during the calcination appears to modify the relationship between consistency and disintegration. Preliminary data suggest that the consistency of stucco containing these admixtures is no longer a direct function of disintegration, but that surface properties play a significant role.

In considering other factors that may influence water demand, many intrinsic stucco properties must be taken into account such as purity, particle size and morphology of the gypsum, calcination methods, and stucco surface properties. In addition, the water demand of stucco is dependent on its history such as aging time and conditions, aridization, and presence of surface active agents.

For example, most stucco specimens contain a significant proportion of insoluble impurities, usually inorganic salts such as anhydrite, calcite, and dolomite. These salts do not disintegrate spontaneously upon dispersion in water and therefore do not increase the water demand. Consequently, the water demand of a given stucco will decrease as the impurity increases.

During the calcination process, the gypsum undergoes comminution caused

TABLE 3—*Terra Alba batch calcined consistency/disintegration regression data.*

Consistency Measurement	Slope, mL/100 g	Intercept, mL/100 g	Correlation
DIN	0.334	74.1	1.000
Pouring	0.681	69.6	0.989
60 s	1.41	363	0.882

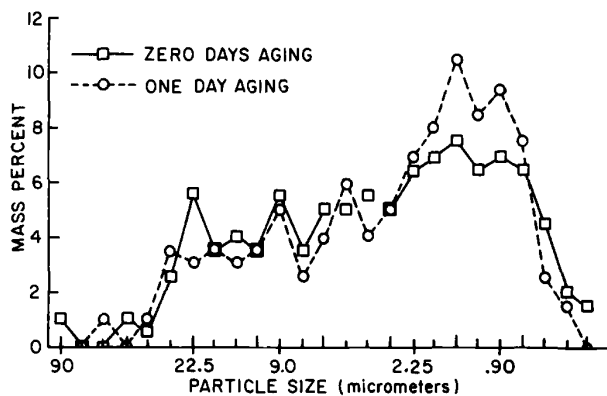


FIG. 7—Comparison of 7-s dispersion zero- and one-day aging.

by both dehydration and stirring/fluidization. With decreasing particle size of the gypsum, the dehydration proceeds more uniformly and generates a more constant water vapor pressure in the kettle. Stucco prepared under these conditions will not disintegrate to the same degree as stucco prepared from larger gypsum particles calcined more rapidly in a less homogeneous environment, and consequently, will tend to have a lower water demand.

The method by which gypsum is calcined may have a profound effect on the water demand of stucco because it dictates the local conditions at the calcining surface. For example, in an autoclave, the gypsum is dehydrated through a solution process. This prevents the formation of soluble anhydrite and yields a stable solid, which has undergone little or no lattice disruption. As a result, the particles have low surface energy. In a tray calcination, the gypsum is rapidly dehydrated in a low water vapor environment. The formation of soluble anhydrite is promoted and lattice disruption and particulate surface energy are increased, resulting in a relatively unstable product. The intermediate case is a continuous

TABLE 4—*Terra Alba* continuously calcined consistency/surface area data.

Consistency Measurement	Disintegration, %	Consistency, mL/100 g	Surface Area, cm <sup>2</sup> /g	Water Thickness, 10 <sup>-6</sup> m
DIN	0	76.2	4 400	17.3
	100	115.8	24 700	4.7
Pouring	0	72.0	4 400	16.4
	100	95.4	24 700	3.9
7 s	0	40.6	4 400	9.2
	100	102.6	24 700	4.2
60 s	0	346	4 400	78.6
	100	470	24 700	19.0

TABLE 5—*Terra Alba* batch calcined consistency/surface area data.

Consistency Measurement	Disintegration, %	Consistency, mL/100 g	Surface Area, cm <sup>2</sup> /g	Water Thickness, 10 <sup>-6</sup> m
DIN	0	74.1	4 000	18.5
	100	107.5	20 400	5.3
Pouring	0	69.6	4 000	17.4
	100	137.7	20 400	6.8
60 s	0	363	4 000	90.8
	100	504	20 400	24.7

kettle in which the gypsum particles are being dehydrated with constant stirring in a nearly saturated steam atmosphere. The formation of soluble anhydrite is inhibited and moderate lattice disruption takes place.

The water demand of a stucco can be reduced through the addition of surface active agents into the gaging water. These surface active agents stabilize the interface between the solid and aqueous phase and can reduce inter-particulate adhesion. This in turn yields a slurry of greater fluidity at a given solids concentration.

The physical, chemical, and electrical properties of the stucco particle surface will affect the affinity of the stucco for the aqueous phase and the resilience of the stucco surface when subjected to external influences such as agitation. These in turn affect the thickness of the solid/liquid interface and the amount of particulate disintegration that can occur, thus affecting the fluidity of the slurry at a given solids concentration.

## Conclusions

The objective of this study was to examine the relationship between water demand and particle size distribution of stucco as affected by changes in disintegration during aging. The results of this work show that both the water demand of a stucco and its disintegration respond to aging in a similar fashion. The experimental data suggest that when all other factors are kept constant, there is a direct relationship between water demand and particle size distribution, which is linear for all practical purposes, and that particle size distribution is governed by disintegration.

In the presence of aridizing agents or surfactants, the consistency is no longer a direct function of only the disintegration but appears to depend on surface properties and particle morphology. This indicates the possibility of obtaining a stucco with a low water demand without loss of high disintegration which is necessary to maintain desired setting and strength properties. Further work is required to elucidate the influence of surface properties and other factors on the interdependent relationship between water demand and particle size distribution.

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