

# Density and strength characteristics of foamed gypsum

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## Abstract

This paper deals with the effect of substances producing and assisting gas and foam formation on the properties of gypsum density and strength. The gas-based foaming method utilises aluminium sulphate, potassium alum or ammonium bicarbonate for the chemical production of gas bubbles in gypsum paste. The second method obtains foamed gypsum by means of air entrainment in wet gypsum paste. The foaming agents chosen were sodium lauryl sulphate and nonyl phenol ethoxylate that are widely employed in detergent production. Five different foaming techniques are obtained with these additives required for gas and foam production. Citric acid, as retarder, and carboxyl methyl cellulose (CMC), as viscosity increasing agent, were used for promoting foam and gas formation during the foaming experiments. Aluminium sulphate was discovered to be the one that achieves the most foaming in gypsum. The techniques utilising potassium alum, sodium lauryl sulphate and ammonium bicarbonate reduced the density of gypsum products by values varying between 30% and 35%. Foaming with nonyl phenol ethoxylate had an insignificant effect on density. The addition of citric acid and carboxyl methyl cellulose assisted with density reduction. Besides regulating the hardening time, citric acid supported density reduction by releasing gas through a reaction with marble powder. However, an addition of CMC above 0.1% or 0.2% causes a density increase by preventing bubbles to expand and merge with each other. The compressive strength of low-density gypsum products falls well below 10 Mpa, stipulated by TS 370 for building gypsum. © 2000 Elsevier Science Ltd. All rights reserved.

**Keywords:** Foamed gypsum; Aluminium sulphate; Potassium alum; Ammonium bicarbonate; Sodium lauryl sulphate; Nonyl phenol ethoxylate; Carboxyl methyl cellulose (CMC); Citric acid

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## 1. Introduction

Gypsum is a material with a relatively small weight per unit volume under normal conditions. To further reduce the density of gypsum, the water ratio is increased or lightweight aggregates such as vermiculite, perlite, expanded polystyrene are added. Beside these methods aeration of gypsum is also widely used. Two methods are commonly used for the production of porous gypsum. The first is based on the principles of forming gas bubbles in gypsum paste through chemical additives. The second method is entrapment of air in wet gypsum paste through whisking and tempering by using additives that reduce the internal surface tensions, resulting in aerated gypsum.

In the first method, it is important that the quality of gypsum is not impaired by the chemical additives. The fact that the calcium sulphate salt present in the material

obtained at the end of the chemical reaction is insoluble in water prevents any degradation of gypsum. Sulphate salts of a weak alkali such as potassium aluminium sulphate (potassium alum), zinc sulphate, copper sulphate, aluminium sulphate additives for reducing the time for hardening or accelerating the reaction of gypsum cause the formation of gas in the mass of gypsum coupled with marble powder [1]. The investigation of the reaction of these materials with marble powder reveals that the final product is synthetic gypsum and can be defined as dehydrate which is the water-insoluble type of calcium sulphate salt. An adverse effect on the quality of gypsum does not occur. The production of porous gypsum is also confirmed to be possible through to the utilisation of synthetic gypsum that is resistant to acid action encountered in the production of hydrofluoric acid [2]. At present, large amounts of phosphogypsum and fluorogypsum are obtained during the production of hydrofluoric and phosphoric acid. A phosphate rock that belongs to the mineral of phosphorous rock salt dissolves in sulphuric acid and water, producing

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phosphogypsum, phosphoric acid and small amounts of hydrofluoric acid [2]. In the air-entraining gypsum production method, soaps [3,4], oil acids [5–11], sulphates [12,13], sulphonates [14–17] and ethoxy groups [18] are added into the mixing water of gypsum. These additives that are called the surface active agents [19] provide for the stabilisation of bubbles and enable aeration [20].

The hardening time is observed to be the basic function of the formation of gas as well as foam in all applications of porous gypsum. Therefore determination is required of the appropriate times for hardening and consequently the type and ratio of the retarder or accelerator to be used. Citric acid, borax, tartaric acid, sodium citrate, lactose, keratin, maltose and saccharose are the most commonly used retarders for gypsum [21]. When citric acid is used as a retarder in gypsum the observations made are the reduction in the number of open pores, the increase of the average pore diameter, the general reduction of small air bubbles in conjunction with the increase of pore diameters, and finally a reduction in the bending and compressive strengths and degree of hardness [2]. The entrapment of gas bubbles in the gypsum paste during the process of gypsum aeration is a factor that is as important as the determination of the setting time. The additives for increasing viscosity prevent the loss of gas and assist the aeration by enclosing the crystals of calcium sulphate in the wet gypsum paste so that the resistance to flow is increased.

The aeration methods described above were used in this research where reduction of weight per unit volume of gypsum was investigated. The effects of the percentages of additives for producing and assisting gas and foam formation and also the effects of the ratio of mixing water in the mixture on the physical and mechanical properties of gypsum are investigated.

## 2. Experimental

### 2.1. Materials and method

Gas-releasing and air-entraining gypsum production methods are used in this work for obtaining porous gypsum. For the gas-releasing method ammonium bicarbonate and sulphate salts such as aluminium sulphate and potassium alum are used as chemical additives while surface active substances such as sodium lauryl sulphate and nonyl phenol ethoxylate are employed as foaming agent in the air entraining method. Thus, five different foaming techniques are obtained with the additives mentioned above. The test matrix is shown in Table 1. These techniques are described as follows:

*Foaming techniques with sulphate salts.* In these techniques, the mixture of gypsum products contains gypsum (Calcium sulphate hemihydrate  $\beta$ ), water, marble

powder and sulphate salts such as aluminium sulphate and potassium alum. Aluminium sulphate and potassium alum were selected on the basis of their properties that they produce dehydrate as an insoluble type of calcium sulphate at the end of the reaction, they improve the mechanical performance and hardness of gypsum undergoing a loss of strength arising from the porous structure and they do not degrade the quality of gypsum. Sulphate salts dissolve in the mixing water. Porosity is achieved by means of carbon dioxide that appears when sulphuric acid, produced due to the dissolved salts, is mixed with marble powder. The temperature of the mixing water added to the gypsum was maintained at 37–38°C as the heat of mixing water aids the dissolution of salts such as aluminium sulphate and potassium alum. Furthermore warm water allows the hardening time of gypsum to be extended and supports the foaming process. Citric acid as a retarder and carboxyl methyl cellulose as a viscosity increasing agent were used to assist gas formation during the foaming experiments. In these techniques, while the contents of aluminium sulphate and potassium alum in the mixture are both held constant at 1%, the content of citric acid varied between 4–6%, CMC 0.1–0.4% and mixing water 55–70%.

*Foaming technique with ammonium bicarbonate.* This technique is based on entraining within pores of the gypsum, carbon dioxide gas released as a consequence of the reaction between ammonium bicarbonate and water. This technique entails the addition of 0.8–2% by weight ammonium bicarbonate to the calcium sulphate hemihydrate. The density was calculated to be minimum when the content of ammonium bicarbonate is 0.12%. On this assumption the same content was applied to all experiments. The contents are varied of citric acid between 0.5–1%, CMC 0.1–0.2% and water 57.5–85%.

*Foaming techniques with surface active agents.* There were no differences between the foaming agents used in these techniques because they are all foreign materials in gypsum. Therefore experiments were carried out using sodium lauryl sulphate and nonyl phenol ethoxylate employed widely in the production of detergent. Gypsum mixtures were prepared with 0.1–0.4% by weight sodium lauryl sulphate powder or 0.1–0.6% nonyl phenol ethoxylate in 70% mixing water. Subsequently the gypsum paste was stirred for 3–5 min in a bidirectional mixing movement with the blades of the mixer held manually at an angle with the horizontal plane. Manual stirring gives rise to the failure of obtaining standard conditions in experiments. This fact brings about the probability of errors in the results. The experimental samples were poured into 70×70×70 mm<sup>3</sup> corrosion-resistant moulds. The samples prepared using surface active agents failed to attain sufficient strength in the laboratory conditions of 22°C air temperature and 60–80% relative air humidity for removing from their

Table 1

Density and compressive strength of gypsum foamed with aluminium sulphate and potassium alum

Chemical additive	Water/ gypsum (%)	Citric acid (%)			CMC (%)				Density (kg/m <sup>3</sup> )	Compressive strength (MPa)
		4	5	6	0.1	0.2	0.3	0.4		
Aluminium sulphate	55	+			+				909	1.24
	55	+				+			929	1.80
	55	+					+		878	1.33
	55	+						+	913	1.42
	55		+		+				852	1.38
	55		+			+			868	1.46
	55		+				+		863	1.31
	55		+					+	870	1.02
	55			+	+				832	0.55
	55			+		+			839	1.12
	55			+			+		918	0.81
	55			+				+	812	1.09
	60	+						+	841	1.10
	60		+					+	762	0.49
	60			+				+	782	0.41
	65	+						+	667	0.88
	65		+					+	694	0.63
	65			+				+	763	0.75
	70	+						+	658	0.41
	70		+					+	712	0.65
	70			+				+	761	0.54
Potassium alum	57.5				+				821	1.20
	57.5					+			809	0.59
	60				+				843	1.65
	60					+			835	0.87
	62.5					+			924	0.99
	65					+			981	1.29

moulds. Therefore the samples were dried for 24 h at 40°C in an oven prior to the density and compressive strength measurements.

The mixtures used for the preparation of samples have different setting times. Consequently these times were measured separately for each foaming technique by ascertaining the initial and final times required for setting. A vicat needle was used for measuring the setting times. According to the Turkish Standard (TS 370), the setting time is stipulated to be not less than 10 min. The times of over 10 min for the initiation of setting and 20–25 min for the final are established as the ideal times for foaming with gas during the preliminary experiments. As for the air-entraining method, a time period allowing workability is sufficient for foaming.

### 3. Results and discussion

#### 3.1. Determination of the additive ratio by establishing the setting time

Criteria affecting the setting time of gypsum, such as the ratio of retarder and the amount of mixing water,

are the principal elements of foamed gypsum production. An insufficient retarder ratio gives rise to a small amount of foaming and also to cracking on the mass of gypsum. Conversely in the case of an excessive retarder ratio the delayed formation of gas bubbles causes sedimentation of the aqueous gypsum paste. If the ratios of foaming and retarding agents are determined appropriately then the foaming of gypsum can be increased. Therefore the retarder ratios providing the necessary time for foaming should be established accurately by determining the setting time. When citric acid in a ratio between 4% and 6% is added to gypsum paste containing 1% aluminium sulphate, as can be observed in Fig. 1, the required time for foaming can be reached. However adding 0.4% of CMC increased the resistance to flow significantly. Thus matrix samples are prepared containing 0.1–0.2% CMC and 57.5–65% mixing water in the technique with 1% of potassium alum. According to the results of the setting time measurements, as shown in Fig. 2, 0.75% citric acid is sufficient for foaming.

Gas is produced in the reaction of ammonium bicarbonate with water. Therefore it cannot be ascertained whether a mixture with gypsum and this chemical accelerates or retards the reaction. Consequently the

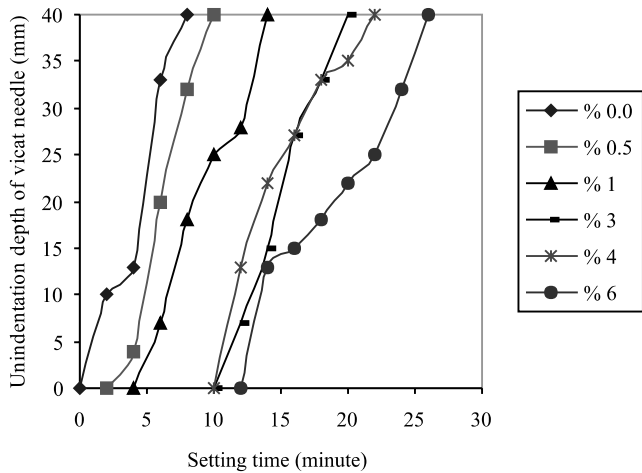


Fig. 1. Setting times of gypsum paste containing 1% aluminium sulphate at various citric acid ratios.

hardening time cannot be determined. For this reason the setting time of pure gypsum with 57.5% mixing water is measured. Fig. 3 indicates that 0.5% citric acid is sufficient for foaming. When the retarder ratio is increased to 1% the hardening is incomplete even after 80 min. Matrix samples with 0.1% and 0.2% CMC-mixing water are prepared for investigating the effects of variations in additive ratios on the density and compressive strength. The samples contain nonyl phenol ethoxylate added to the mixing water in ratios varying between 0.1% and 0.6%. The necessary retarder ratio for foaming these samples was determined to be 0.5% through the process of establishing the hardening time. This ratio is revealed to be 0.2% for the gypsum samples foamed with sodium lauryl sulphate which is added to the mixing water of gypsum in the ratios varying between 0.1% and 0.4%. Gypsum acquires a porous structure in these foaming techniques entailing surface active agents. 0.5–0.7% potassium alum is added to gypsum for increasing the reduced compressive strength.

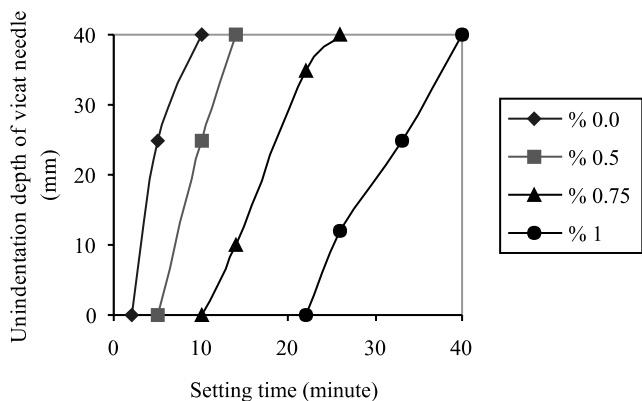


Fig. 2. Setting times of gypsum paste containing 1% potassium alum at various citric acid ratios.

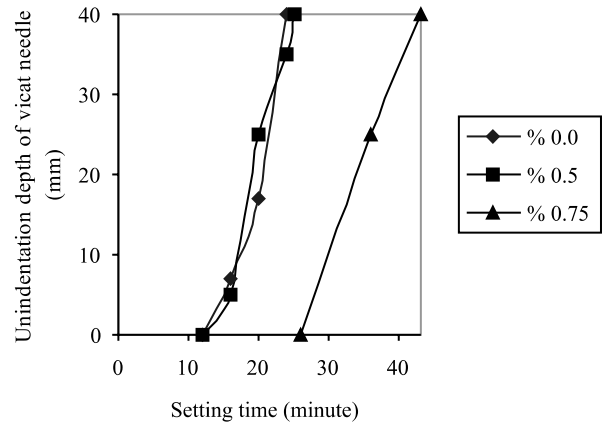


Fig. 3. Setting times of gypsum paste with 57.5% mixing water at various citric acid ratios.

### 3.2. Variation of density according to additives

The effects of additives were investigated by analysing the density variations of samples in the content of retarder-viscosity and retarder-water matrices. For this experiment, 55% mixing water and 1% aluminium sulphate were used. The effects of different percentages of CMC and citric acid on density are discussed below. The density of the samples with 0.1, 0.2 and 0.4% CMC decreases with an increase in the percentage of citric acid, as apparent in Fig. 4 showing the relationship of retarder-density. However a concentration of density is observed at 0.3% CMC attributed to the error in the density value of the sample with 0.3% CMC and 6% citric acid. In general density decreases as the retarder ratio increases, confirming that citric acid releases carbon dioxide gas by reacting with the marble powder. The non-homogeneity of the curves may arise from the fact that a standard environment is not achieved during the casting of experiment samples. Minimum density is

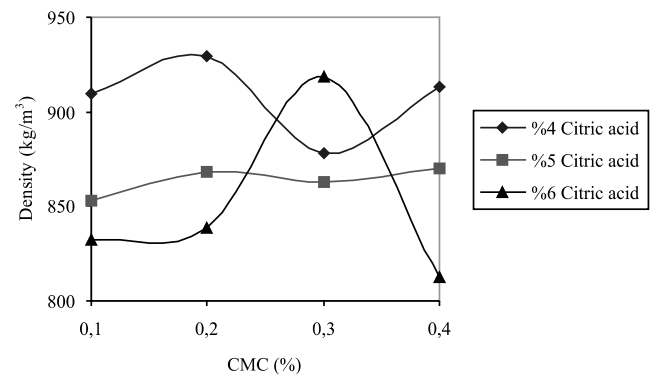


Fig. 4. Effect of viscosity increasing agent content on the density of gypsum foamed with aluminium sulphate at various citric acid ratios.

reached for the sample with 6% citric acid and 0.4% CMC. This fact confirms the correctness of the ideal setting times that are previously proposed in the preliminary experimental studies, as indicated on the hardening time graph (Fig. 1). The initial setting time was specified as more than 10 min and the final setting time as between 20 and 25 min.

The graph of the relationship between viscosity and density demonstrates that the latter is not significantly influenced and increases in conjunction with an increase of the CMC percentage (Fig. 5). This rise in the weight per unit volume may be attributable to the fact that 55% water content is insufficient for foam generation at high CMC ratios. Also during the casting of samples foaming becomes difficult when the viscosity is increased. The achievement of minimum density at 0.4% CMC and 6% citric acid may arise from the increased content of citric acid rather than intensive foaming of CMC. The effects on variation of density were investigated from the rise in mixing water percentage at different retarder ratios since 55% mixing water remains insufficient for satisfactory foaming as the CMC ratio is increased.

The graph of the relationship between mixing water and density reveals that the density diminishes as the water content increases at ratios of 55–60% (Fig. 6). A rise in the water/gypsum ratio for a plastic gypsum paste prolongs the setting time. However, since this prolonging effect of the mixing water is further accentuated by citric acid, the delayed gas bubbles may give rise to sedimentation in the gypsum paste that is still not set. The occurrence of a rise in density at all water percentages above 65% of mixing water may be caused by the sedimentation inside the aqueous gypsum paste, as demonstrated by Fig. 7. The same cause may also account for obtaining minimum density for the sample with 70% mixing water and 4% citric acid, while the sample with 55% water and 6% citric acid is at minimum density, as indicated by Fig. 7.

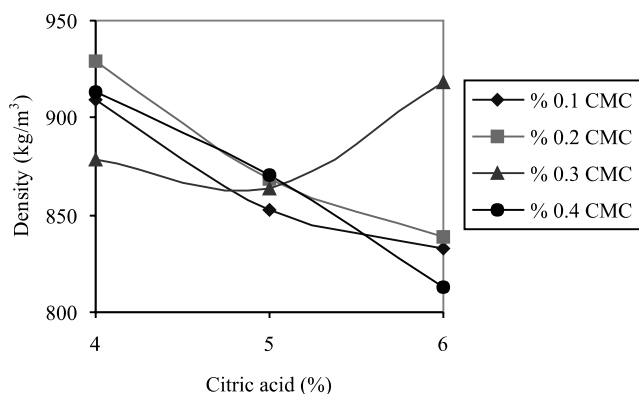


Fig. 5. Effect of retarder content on the density of gypsum foamed with aluminium sulphate at various CMC ratios.

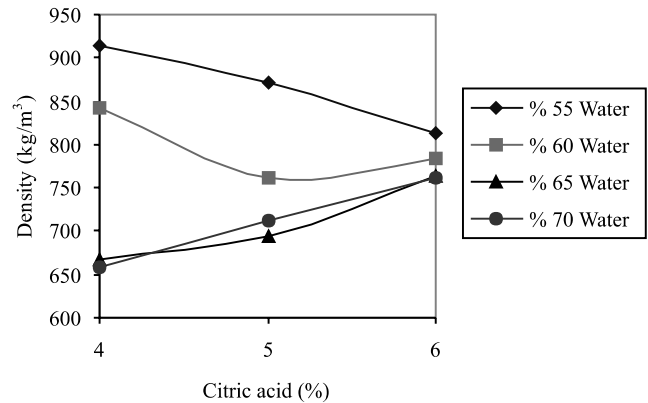


Fig. 6. Effect of retarder content on the density of gypsum foamed with aluminium sulphate at various water contents.

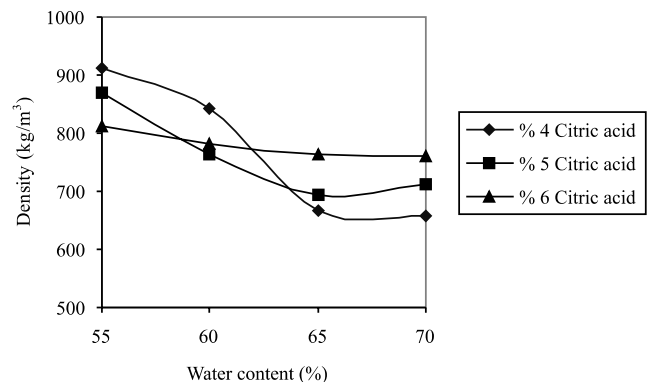


Fig. 7. Effect of water content on the density of gypsum foamed with aluminium sulphate at various citric acid ratios.

The effects of the additives on density variation were also investigated for the samples foamed with potassium alum in a matrix of CMC and mixing water (Table 1). The reduction in density when viscosity is increased may be caused by more foaming in a matrix where the resistance to flow is higher. Density increases with the water content. This may be a result of the fact that gas bubbles in aqueous gypsum paste escape easily outside as the water content increases.

The investigation of the density variation in the matrix of CMC and mixing water containing 0.5% citric acid and 0.12% ammonium bicarbonate reveals that density diminishes when the water content is increased at constant CMC percentages (Table 2). Conversely when the water content is held constant and the CMC percentages are increased, it is observed that density diminishes up to 62.5% mixing water and increases above this percentage of water. When the retarder ratio is increased from 0.5% to 1%, while the ratio of ammonium bicarbonate is retained at 0.12% as constant, density is observed to increase in general, yielding differences of between 20 and 80 kg/m<sup>3</sup>. However, when

Table 2

Density and compressive strength of gypsum foamed with ammonium bicarbonate

Water/ gypsum (%)	Citric acid (%)		CMC (%)			Density (kg/m <sup>3</sup> )	Compressive strength (MPa)
	0.5	1	0	0.1	0.2		
57.5	+		+			1086	2.22
57.5	+			+		1040	1.60
60	+		+			1010	1.97
60	+			+		972	1.34
62.5	+		+			980	1.93
62.5	+			+		972	1.25
62.5	+				+	1024	1.82
65	+		+			925	1.69
65	+			+		962	0.85
65	+				+	972	1.24
62.5		+	+			1060	
62.5		+		+		1026	
62.5		+			+	1000	
65		+	+			1020	
65		+		+		978	
65		+			+	946	
70	+				+	850	0.95
75	+				+	780	0.63
80	+				+	770	0.48
85	+				+	756	0.35

0.5% of citric acid is added, the density decreases as water content is increased while viscosity is constant. Similarly for the same amount of citric acid density decreases as water content is constant while viscosity is increased. The same principle applies when 1% citric acid is added to the gypsum paste. The reason for the general increase of density with any increase of citric acid might be the prolonged hardening time. Any rise in the viscosity ratio does not affect the fall in the density to a great extent, as given by Table 2. An increased water content reduces the weight per unit volume by enabling the gas bubbles in the plastic gypsum paste to expand. To this end, variation of density is explored at 0.2% CMC and 0.12% ammonium bicarbonate kept as constant, while mixing water percentage is increased. A reduction in the weight per unit volume down to 756 kg/m<sup>3</sup> is observed to be possible.

A foamed product with a density 821 kg/m<sup>3</sup> can be obtained by whisking gypsum paste containing calcium sulphate hemihydrate  $\beta$  to which is added by weight 0.2% citric acid retarder and 0.1% sodium lauryl sulphate (Table 3). The weight per unit volume falls to 746 kg/m<sup>3</sup> when sodium lauryl sulphate content is increased to 0.3%. When 0.5% and 0.7% potassium alum are added at a constant ratio of 0.4% sodium lauryl sulphate different values of densities such as 683 kg/m<sup>3</sup> and 708 kg/m<sup>3</sup> are obtained. This deviation may be due to the failure of establishing standardised conditions in the experiments. A foamed product with a density of 1093 kg/m<sup>3</sup> is obtained by adding by weight 0.5% citric acid

and 0.1% nonyl phenol ethoxylate into calcium sulphate hemihydrate  $\beta$  (Table 3). When the percentage of nonyl phenol ethoxylate is raised to 0.3%, the weight per unit volume is reduced to 857 kg/m<sup>3</sup>. Adding by weight 0.4% nonyl phenol ethoxylate and 0.5% potassium alum to the gypsum mixture results in a density of 872 kg/m<sup>3</sup>. Similarly, an addition of 0.6% nonyl phenol ethoxylate together with 0.6% potassium alum yields a different density of 828 kg/m<sup>3</sup>. The inconsistency in the final two statements above is likely to arise from the lack of a standardised environment for the experiments.

### 3.3. Variation of compressive strength according to additives

Figs. 8 and 9 display the variation of compressive strengths for the gypsum samples foamed with aluminium sulphate according to the additives used in the foaming process. Compressive strengths are observed to be reduced as the citric acid ratios are increased in the matrix of retarder-viscosity. This reduction in strength is caused by the fact that the gypsum product acquires a porous structure due to the diminished density with increased ratio of retarder. The curves of the viscosity-compressive strength relationships appear to be non-homogenous and irregular because of the lack of a standardised experimental set-up. However, it can be stated that 5% citric acid and 0.1–0.2% CMC do not decrease the compressive strength significantly unlike 0.3–0.4% CMC which does impair this strength. Com-

Table 3

Density and compressive strength of gypsum foamed with sodium lauryl sulphate and nonyl phenol ethoxylate

Foaming agent	Foaming agent content (%)	Potassium alum (%)				Density (kg/m <sup>3</sup> )	Compressive strength (MPa)
		0	0.5	0.6	0.7		
Sodium lauryl sulphate	0.1	+				821	0.76
	0.2	+				780	0.76
	0.3	+				746	1.43
	0.4		+			683	1.50
	0.4				+	708	0.44
Nonyl phenol ethoxylate	0.1	+				1093	1.96
	0.2	+				897	0.86
	0.3	+				857	0.82
	0.4		+			872	1.48
	0.6			+		828	0.49

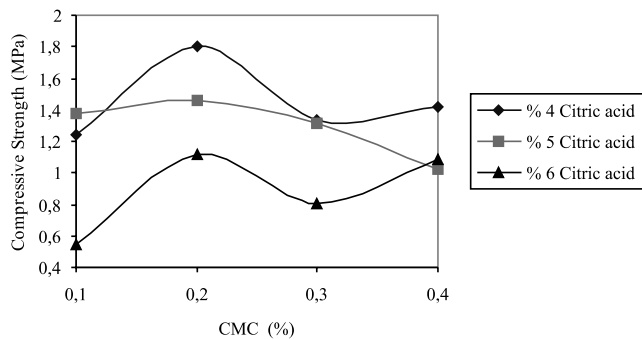


Fig. 8. Effect of viscosity increasing agent content on the compressive strength of gypsum foamed with aluminium sulphate at various citric acid ratios.

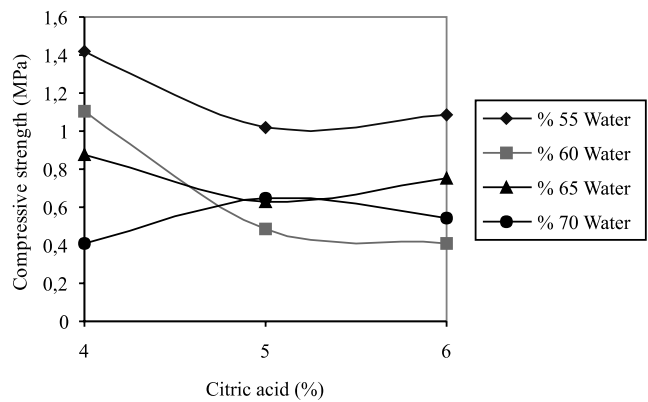


Fig. 10. Effect of retarder content on the compressive strength of gypsum foamed with aluminium sulphate at various water contents.

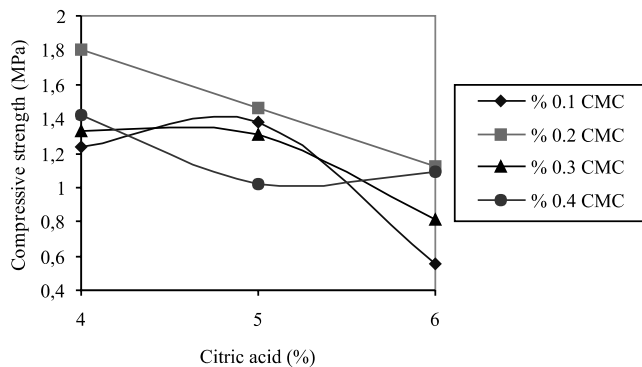


Fig. 9. Effect of retarder content on the compressive strength of gypsum foamed with aluminium sulphate at various CMC ratios.

ments are made below related to Fig. 10 demonstrating the variation of strength as the ratio of mixing water is held constant while the citric acid percentage is raised. An increase of strength is observed at percentages above 5% citric acid and 60% mixing water while a decrease occurs at lower values. The reason for the reduction in

strength is the prolonged hardening times due to the increase in the ratio of citric acid and mixing water and consequently the reduction of density resulting from sedimentation inside the aqueous gypsum paste. Those samples with high viscosity ratios such as 0.4% provided by CMC are more inclined to foaming as the water ratio is increased. Consequently the reduction in their strength can be attributed to their diminished density. The technique in which 1% potassium alum is used, compressive strength changes are directly proportional to density (Table 1). Also in the case of porous samples foamed with ammonium bicarbonate, their compressive strengths are proportional to respective densities. The variations of compressive strength are explored for the CMC-mixing water matrix containing 0.5% citric acid and 0.12% ammonium bicarbonate. Compressive strengths decrease in parallel to densities as water content is increased for constant CMC percentages (Table 2). However, it can be stated in general that the compressive strength decreases as the CMC ratio increases while the water percentage is constant. The

compressive strength is investigated for calcium sulphate hemihydrate  $\beta$  foamed with 0.12% ammonium bicarbonate and also containing 0.2% CMC. The strength is 1.82 MPa at 0.2% CMC and 62.5% mixing water. However as the percentage of water is raised to 85% the strength falls to 0.35 MPa in line with the fall in density. The fact that calcium sulphate hemihydrate  $\beta$  without any addition of CMC attains more compressive strength indicates that CMC reduces strength. A reduction of compressive strength is anticipated in line with density as the percentage of sodium lauryl sulphate in the gypsum paste is increased. However in actual fact the reverse occurs, i.e. strength increases (Table 3). This phenomenon may be explained either by the increased hardness of gypsum by the foaming agent or by the non-homogenous whisking rate.

Compressive strength diminishes in line with density as the percentage of nonyl phenol ethoxylate added to the gypsum paste increases. The shortening in lengths is reduced to the samples foamed with nonyl phenol ethoxylate when subjected to loads during mechanical tests. Simultaneously the deformed pores adhere onto each other thus restoring their compressive strength and finally scattering by sudden breakage. Table 3 reveals that due to non-homogenous data it becomes difficult to assert whether potassium alum increases or decreases the compressive strength of gypsum samples foamed with sodium lauryl sulphate and nonyl phenol ethoxylate.

#### 4. Conclusions

Five different foaming techniques were used for preparing samples. For each technique samples with the minimum density were taken into consideration. The technique with aluminium sulphate was discovered to be the one that achieves the most foaming in gypsum. The techniques utilising potassium alum, sodium lauryl sulphate and ammonium bicarbonate reduced the density of gypsum products by values varying between 30% and 35%. Foaming with nonyl phenol ethoxylate has an insignificant effect on density. Citric acid and CMC were

used in order to promote the foaming process during the experiments. The addition of these two substances gives rise to density reduction. Besides regulating the hardening time, citric acid supports density reduction by releasing gas through a reaction with marble powder. However an addition of CMC above 0.1% or 0.2% causes density increases, by preventing bubbles from expanding and merging with each other. The compressive strength of low-density gypsum products falls well below 10 N/mm<sup>2</sup> that is stipulated by TS 370 for building gypsum. This fall may be prevented by establishing the appropriate proportion in mixtures for substances that initiate and promote growth of bubbles, by adding substances improving mechanical performance and also by providing standardised conditions for experimentation.

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