

THE USE OF WASTE GYPSUM IN MANUFACTURING CALCIUM SILICATE BOARDS BASED ON FLOW-ON PROCESS AND AUTOCLAVE METHOD

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ABSTRACT

This paper presents an investigation on the use of waste gypsum to manufacture calcium silicate boards based on the flow-on and autoclave method at Hiep Phu Company (HPC). The mix proportion, including the percentage of Calcium and Silicate was been changed when adding the waste gypsum. To ensure that the specific reaction processes still be able to create Tobermorite ($\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$) and Xonolite $\text{Ca}_6\text{Si}_6\text{O}_{17}(\text{OH})_2$ for the new product, the waste gypsum sample was first analysed under XRF in order to define the chemical components before using. The XRD and physical tests (bending strength, density, water absorption, drying shrinkage) shows that the products met the classification Type B, Grade III as to -ASTM C1186-08 Standard.

The good test results on the use of waste gypsum in manufacturing Calcium Silicate Board opens a promising approach in using recycled waste at HPC. It has been an important result consolidating tactic objectives of the company to produce high quality products with reasonable costs. Moreover, it gets in line with the demand of customer using green products- friendly with the environment.

KEYWORDS:

Waste gypsum, Flow-on process, Tobermorite, Autoclave (hydrothermal)

INTRODUCTION

In process of making ceiling tiles, gypsum boards are cut from the original dimension (9mm thickness*1220width*2440length) into small boards (9mm thickness*603width*1210length). During this process the large amount of dust and trimming is created. Normally this dust and trimming is considered as waste, but based on research we considered to take this waste gypsum and recycle it in the calcium silicate board production line.

In the Flow-on process, a slurry with high solid concentration flows onto the felt from the head box, the vacuum system removes excess water from the layers, and the rolled up layers are collected by the Forming Roll to make the Green Sheet.

The autoclave (hydrothermal) process is more commonly used in production of building materials such as calcium silicate board, fiber cement, lightweight concrete, etc. In this paper, the calcium silicate board was processed in hydrothermal condition at temperature 178°C , pressure 0.9MPa in a 16 hour cycle as follows: heat-up to 178°C in 4 hours, stable 178°C in 8 hours and release to atmosphere in 4 hours. During this process chemical reactions happened to create Tobermorite $-\text{Ca}_5\text{Si}_6\text{O}_{16}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ and an excess of Quartz – SiO_2 . Adding gypsum accelerated Tobermorite formation in the autoclave (hydrothermal) process

(Kunio Matsui – 2012) and changing CaO/SiO₂ ratio also effected more Tobermorite formation (Do Quang Minh – 2005).

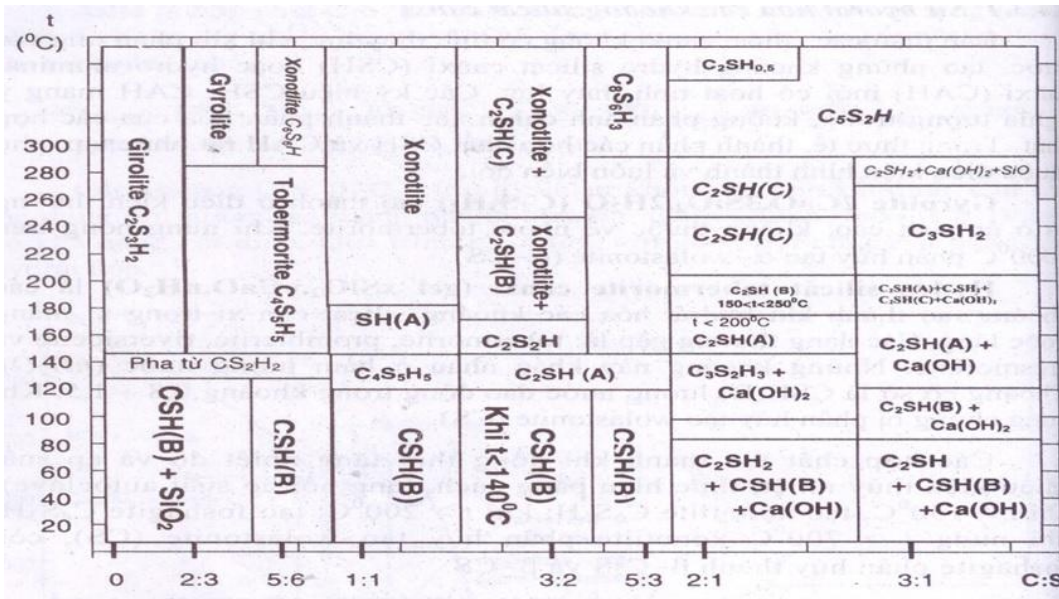


Fig 1: Effect of temperature and C/S ratio for crystal formation in hydrothermal condition[2]

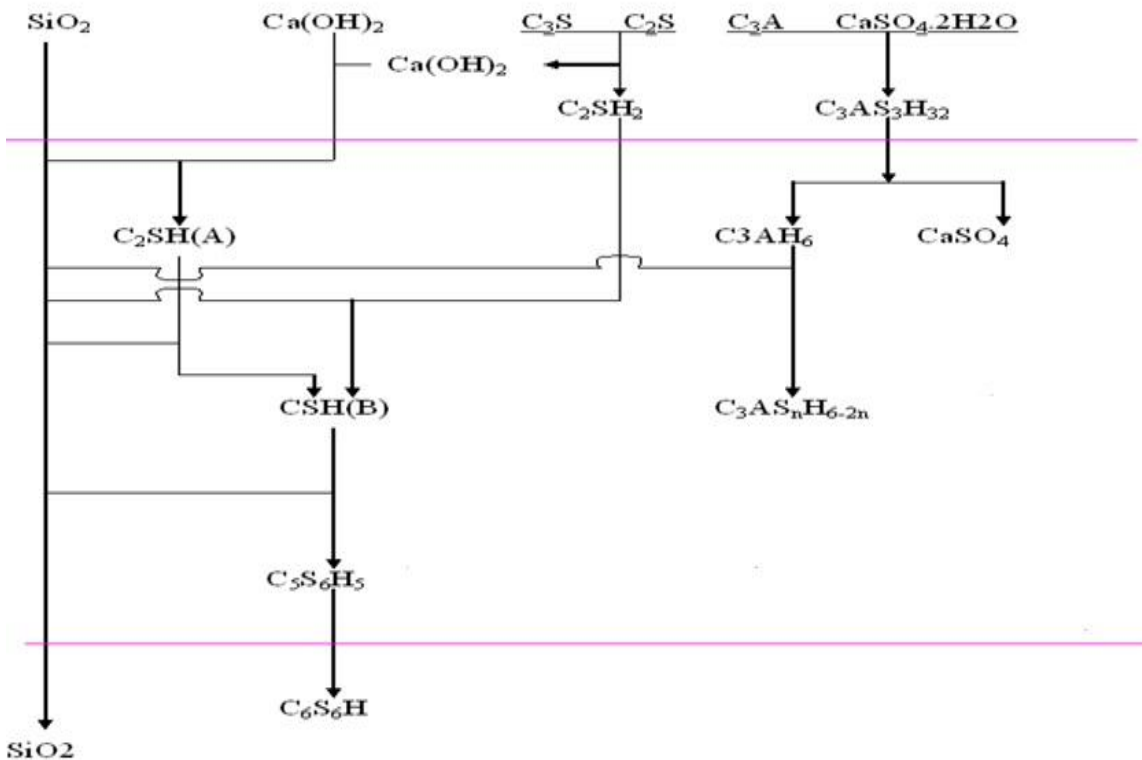


Fig 2:Diagram of tobermorite and xonotlite creation [2]

EXPERIMENTAL

Raw material using:

1. Silica sand: 97.8% purity (SiO₂crystal)
2. Lime stone: provide 70% CaO.
3. Cement PCB40 provide minerals C₃S(3CaO.SiO₂), C₂S(2CaO.SiO₂), C₄AF(4CaO.Al₂O₃.Fe₂O₃), C₃A(3CaO.Al₂O₃).
4. Waste Gypsum : provide 91.72% CaSO₄.2H₂O and 31.7% CaO
5. Pulp to ensure the strength and flexibility of the product.

Waste gypsum was sent to third party laboratory “Quality assurance & Testing center 3” to check the chemical composition by use of XRF according to standard ASTM C471 in order to check the percentage of CaSO₄.2H₂O. The results are shown in the table below.

No	Norms	Unit	Results	Test method
1	CaO	%	31.7	XRF
2	SO ₃	%	43.3	
3	SiO ₂	%	0.62	
4	Al ₂ O ₃	%	0.36	
5	MgO	%	0.19	
6	Combine water	%	19.2	ASTM C471
7	CaSO ₄ .2H ₂ O	%	91.72	
8	CaSO ₄	%	1.08	

Table 1: Result of waste gypsum analysis

Experiment method:

The research is based on the current production line with Flow-on and Autoclave process (hydrothermal treatment). The experiment is based on 3 formulations with different material use. Each formulation produces 300 pieces of dimensions 3.5mm thickness – 1220 width – 2440 length.

Items	Formulation 1	Formulation 2	Normal formulation
Raw materials	4% Waste Gypsum	4% Waste Gypsum	-
	4% Limestone	-	4% Limestone
	Silica sand	Silica sand	Silica sand
	Cement	Cement	Cement
	Pulp	Pulp	Pulp
	Water	Water	Water
CaO/SiO₂(C/S) Ratio	0.477	0.448	0.471

Table 2: Formulation of experiment

All the materials used during the experiment were weighed by weight scale. The compound was mixed to a slurry with 15% dry solids (water to solid ratio is 0.85 by weight, the mixing time is 10 min).

Layer thickness of the film on flow-on process was 0.75 mm and 5 layers (rolls) per sheet.

The sheets were compressed in Stack press 1 with 160 pcs/stack and then pre-curing 3-4 hours at the temperature of 37-45°C after pressing.

The green sheets were then placed in the autoclave and put through the following cycle: heat up 4 hours from 32°C - 178°C - stable in 8 hours at 178°C pressure 0.9 MPa - release pressure from 0.9 MPa to atmosphere in 4 hours.

After the autoclave products were dried in the dryer at a temperature of 60°C in order to assure the moisture content of 10% or less.

Finally the sheets were taken for testing, dimension of sample 152 mm width * 305 mm length following ISO 2859-1. Sample testing is physical test following ASTM C1185 in our own laboratory. Another sample was sent to Analysis center for XRD analysis.

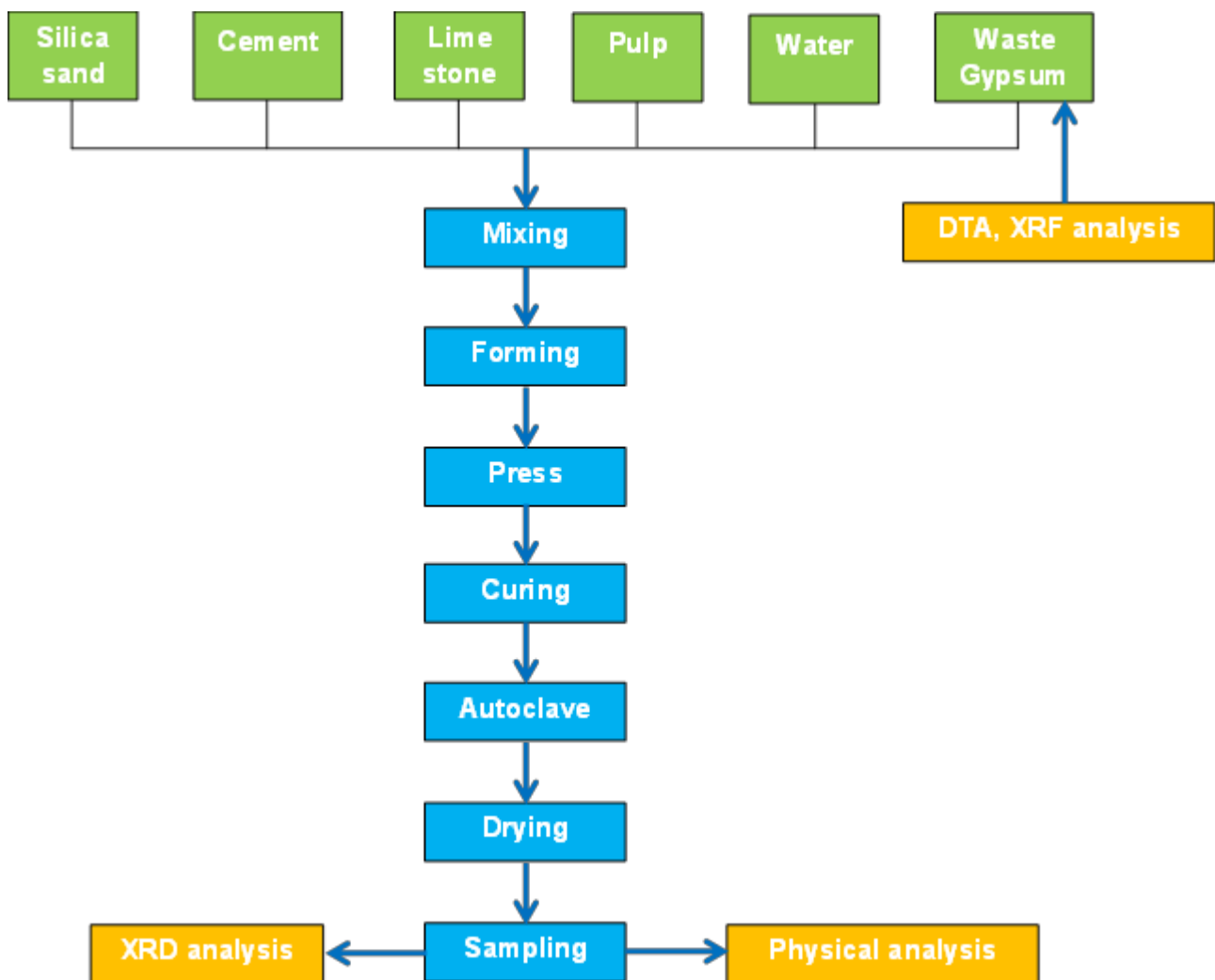


Fig 3: Process of experiment

RESULT AND DISCUSSION

XRD analysis

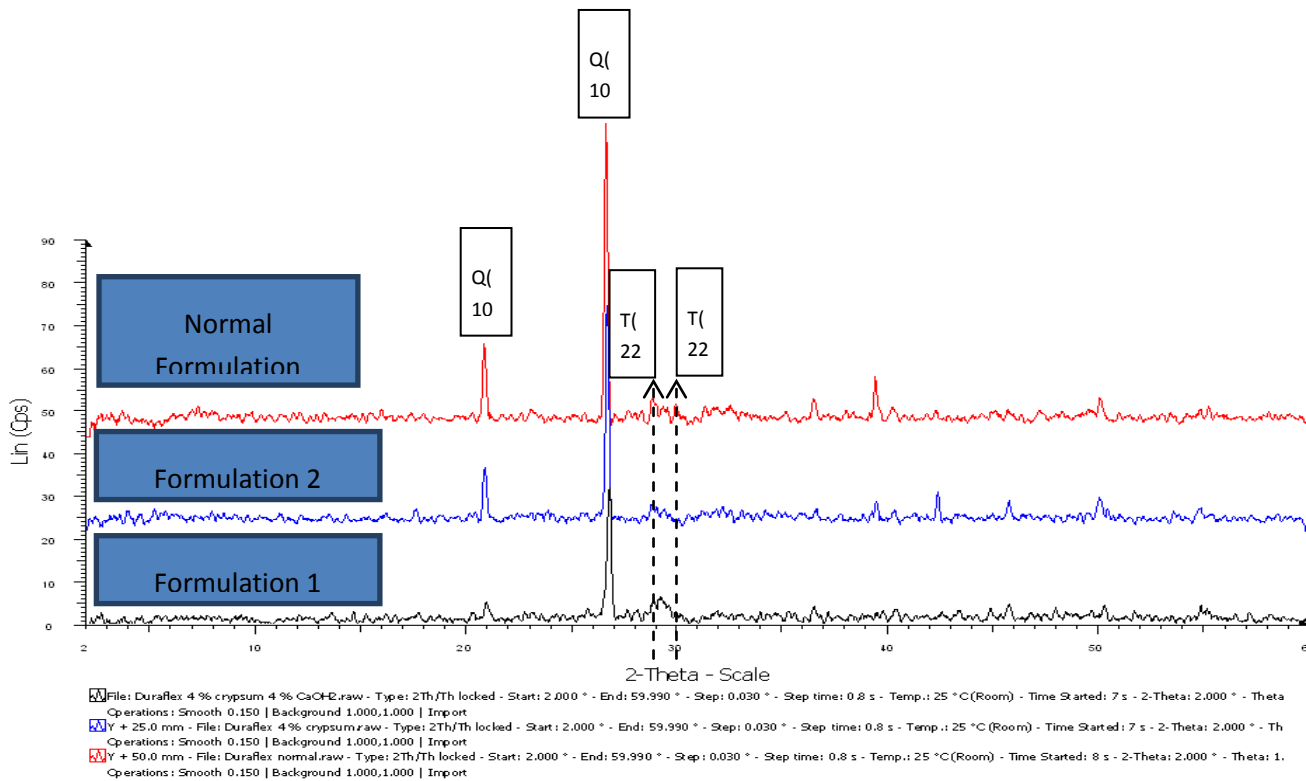


Fig 4: Tobermorite and excess Quartz formation of 3 formulations. T: tobermorite , Q : quartz

Tobermorite formation:

The phase percentage of Tobermorite and Quartz was calculated by following equation of L. Zhang, N.

Hanagata, M. Maeda, T. Minowa, T. Ikoma, H. Fan and X. Zhang, “Porous Hydroxyapatite and Bi-phasic Calcium Phosphate Ceramics Promote Ectopic Os-teoblast Differentiation from Mesenchymal Stem Cells,”

$$T(\%) = \frac{I_{220} \text{ of } T}{I_{220} \text{ of } T + I_{101} \text{ of } Q}$$

I220: Intension of (220) diffraction peak of Tobermorite

I101: Intension of (101) diffraction peak of Quartz

Items	Formulation 1	Formulation 2	Normal formulation
T%(TOBERMORITE %)	21.4%	-	8.6%

Table 3: Result of Tobermorite phase percentage

Table 3 shows formulation 1 with 21.4% tobermorite formation percentage and it is highest T% in 3 experiment's formulations. In formulation 2 we can not calculate the T% because the tobermorite formation was not clear and normal formation shows T% as 8.6%.

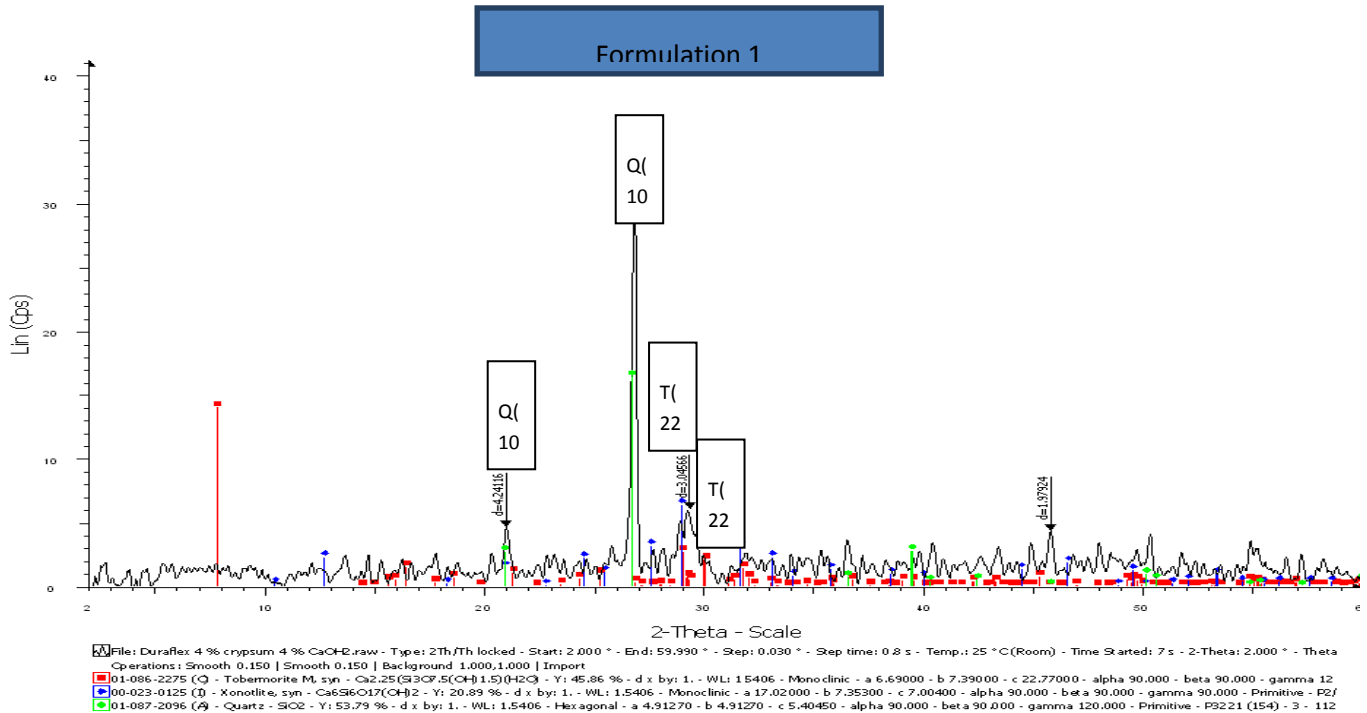


Fig 5: XRD of formulation 1 – T: tobermorite, Q: quartz

Fig 5 (Formulation 1, 4% Waste Gypsum & 4% Lime stone) with the peak intensity T(220) high & sharp, clearly shows Tobermorite formation. The peak intensity T(222) not so high but also demonstrates the presence of Tobermorite crystals. The peak intensity Q(100) and Q(101) is lower than formulation 2 and normal formulation which means less excess quartz in formulation 1.

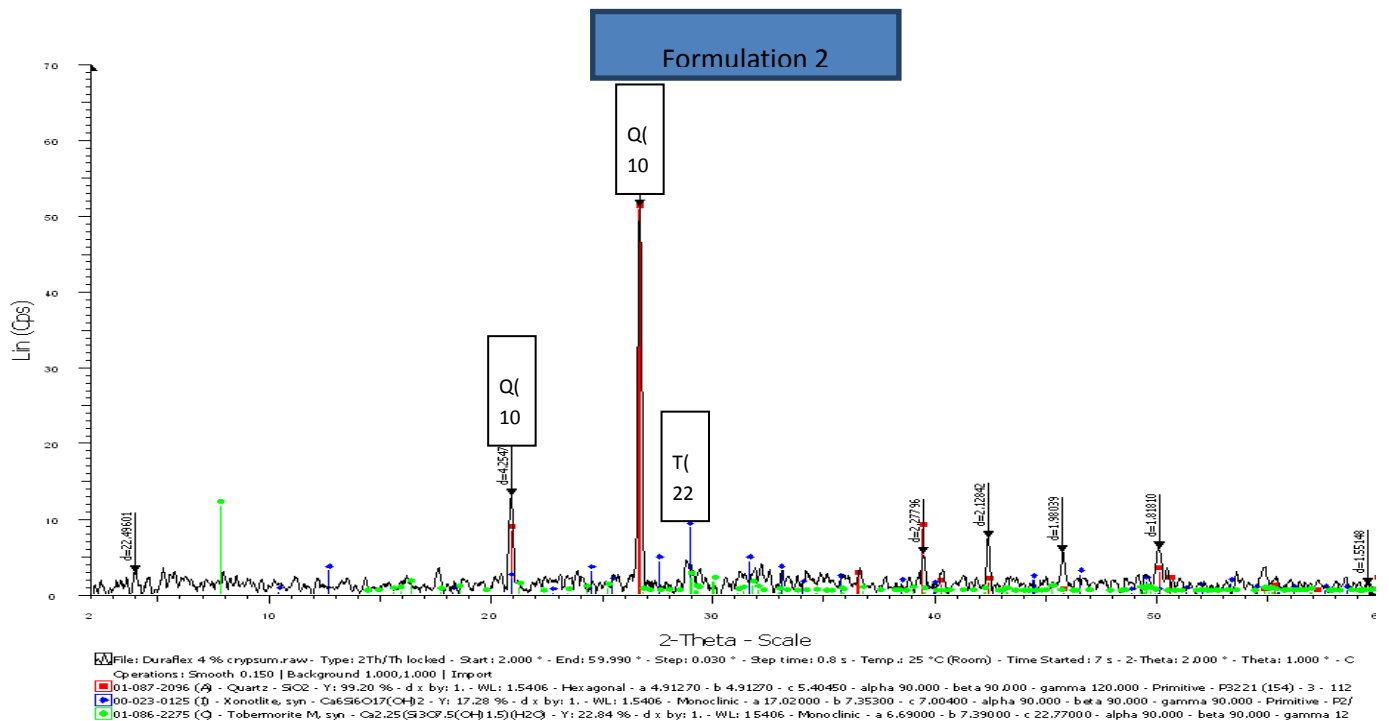


Fig 6: XRD of formulation2 – T: tobermorite, Q: quartz

Fig 6 (Formulation 2, 4% Waste Gypsum without Limestone) shows the peak intensity T(220) is low and low Tobermorite formation. The peak intensities Q(100) and Q(101) are high, showing a large excess of quartz and poor or no Tobermorite crystallisation. Low C/S ratio without Limestone produced less Tobermorite formation in the autoclave.

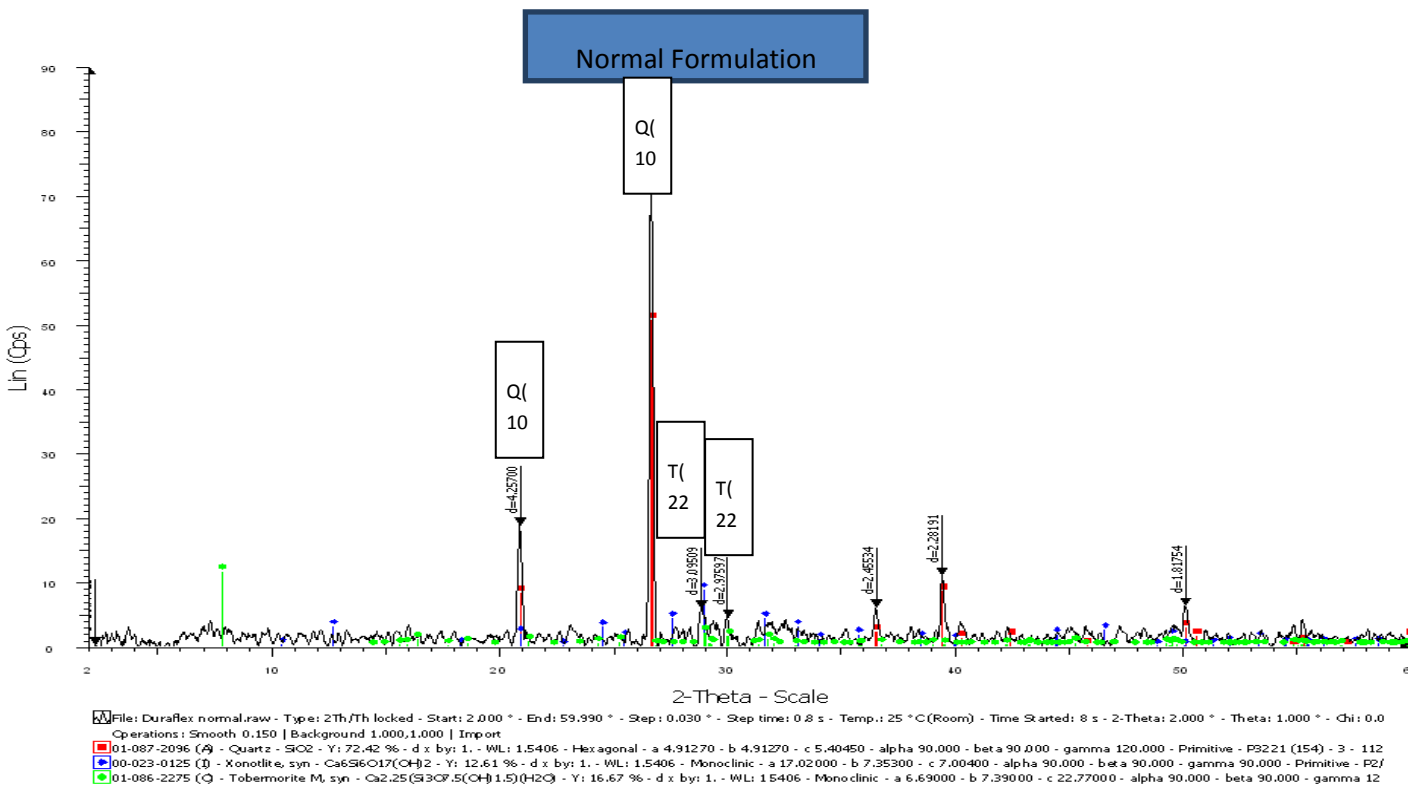


Fig 7: XRD of normal formulation– T: tobermorite, Q: quartz

Fig 7 (Normal formulation without Waste Gypsum & 4% Limestone) shows high and sharp peak intensity T(220), T(222), that shows clearly the Tobermorite formation. As before the peak intensity Q(100) and Q(101) is high, showing a large excess of quartz.

Physical test result:

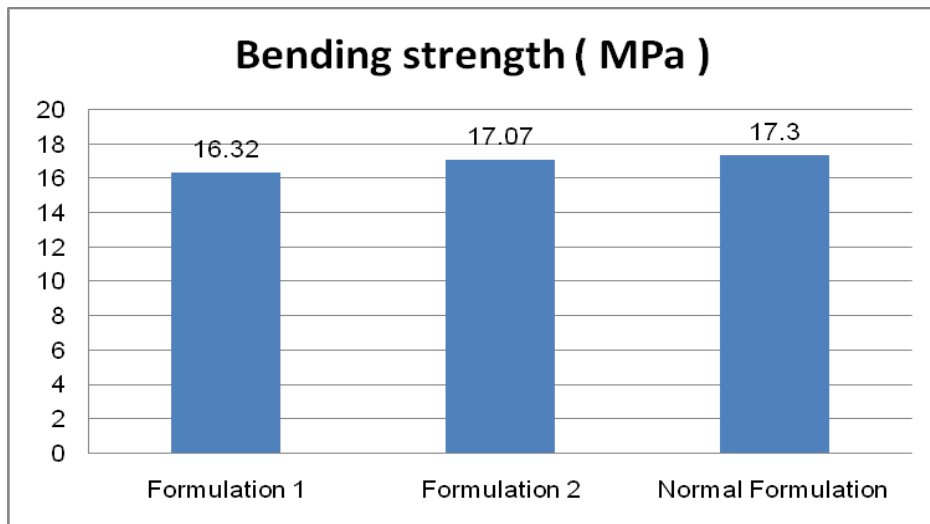


Fig 8 :Bending strength

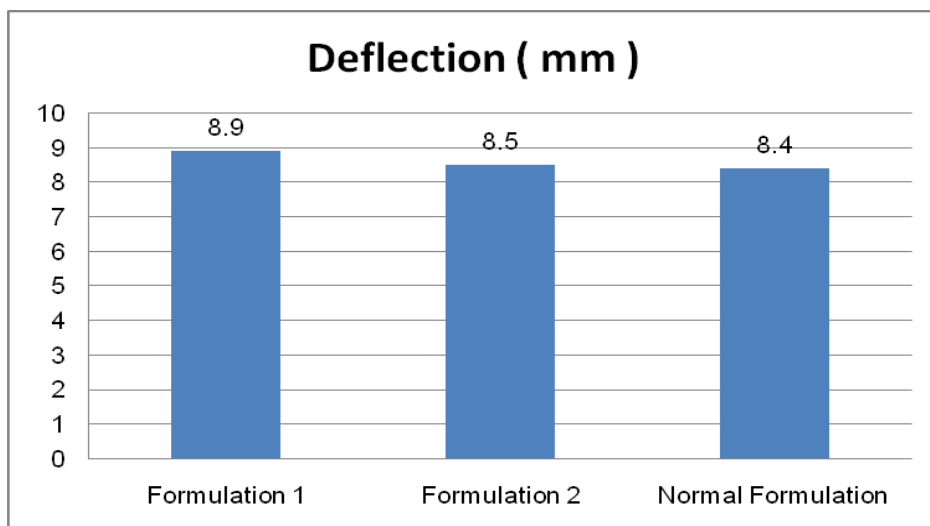


Fig 9: Deflection

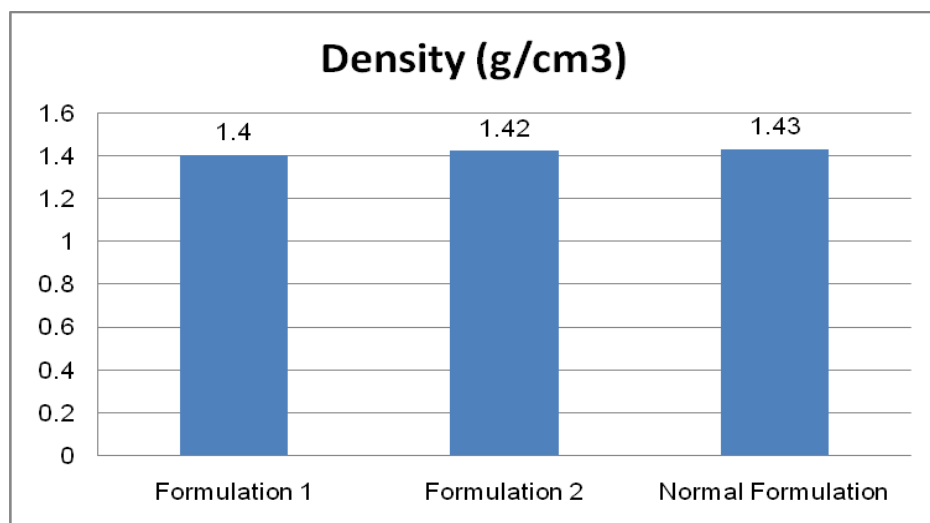


Fig 10: Density

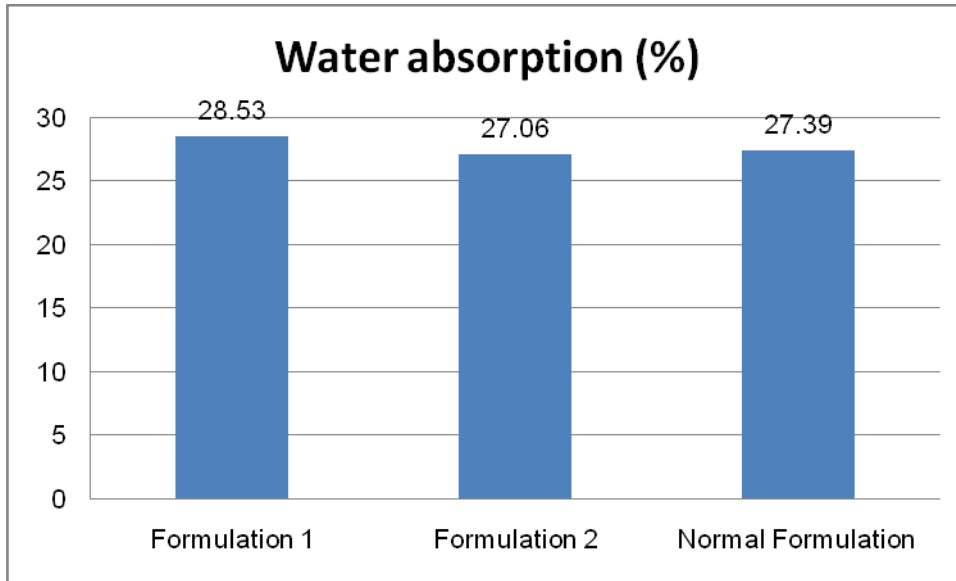


Fig 11: Water absorption

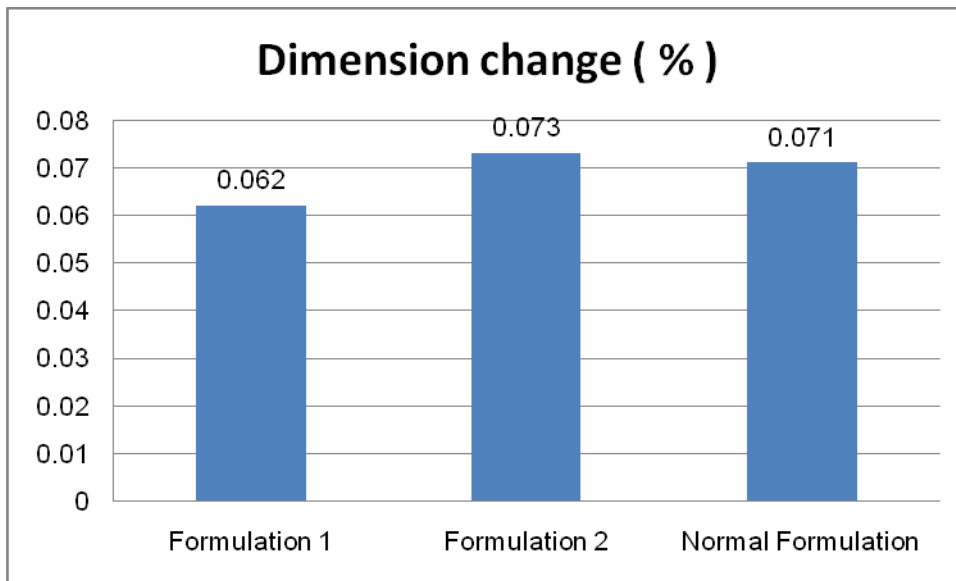


Fig 12: Dimension change

All the physical testing follows ASTM

Bending strength: Bending strength of formulation 1 is 16.32 MPa, lower than formulation 2 and normal formulation being respectively 17.07 MPa & 17.3 MPa. But the bending strength is 16.32 MPa is acceptable for ASTM standard and current company's standard.

Deflection: Deflection of formulation 1 is 8.9 mm, higher than formulation 2 and normal formulation as 8.5mm & 8.4mm. High deflection indicated the product more flexible.

Density: Density of formulation 1 is 1.4 g/cm³, lower than formulation 2 and normal formulation as 1.42 g/cm³ & 1.43 g/cm³. In formulation 1 we have used 4% waste gypsum and limestone, these materials have lower density than the other materials in normal formulation and reduced the product density accordingly.

Water absorption: Water absorption of formulation 1 is 28.53%, higher than formulation 2 and normal formulation being respectively 27.06% & 27.39%. Water absorption is 28.53% is acceptable for ASTM standard and current company's standard. And normally lower density of the product will be higher water absorption.

Dimension change: Dimension change of formulation 1 is 0.062%, lower than formulation 2 and normal formulation being respectively 0.073% & 0.071%. Dimension formulation 1 is good because this formulation uses 4% waste gypsum and limestone, produces more Tobermorite crystal with a high CaO/SiO₂ ratio giving a lower dimension change.

CONCLUSION

Based on the result of physical tests and the XRD analysis in this research, we can see the effect of waste gypsum addition in the flow-on and autoclave process. We can conclude that using waste gypsum with limestone, silica sand, cement, pulp and water made better quality product due to higher Tobermorite crystal formation in autoclave. This product is acceptable to the ASTM standard demonstrating better physical properties such as density, dimension change, deflection, bending strength and water absorption.

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