THE INFLUENCE OF PRECURING BEFORE AUTOCLAVING ON THE PROPERTIES OF FIBRECEMENT SHEETS

DR BEAUTY MTSWENI

Everite Building products, Heidelberg Road, Kliprivier, South Africa

ABSTRACT

In this paper, mechanical properties, microstructure and mineralogical analyses of precured fibre cement sheets before autoclaving were investigated. Microstructure analyses were conducted using scanning electron microscope (SEM) and Quantitative X-ray diffraction (XRD). The work centred around optimising precuring conditions to enhance green strength development of sheets so as to be able to lift the sheets from the form plates onto the autoclave carriers without damage. The early strength is contributed by the hydration of tricalcium silicate in cement.

The observations indicated that shrink-wrapping the sheets at room temperature for 24 hours gave superior properties than unwrapped sheets. The longer the duration of sheets in the precuring chamber, the decline in its mechanical properties was noted. The formation of additional CSH phases, such as tobermorite and hydrogarnet, significantly enhanced the mechanical properties of sheets. It was concluded that the precuring process can be optimized either by shrink-wrapping for 24 hours at room temperature or precuring in the chamber for 8 hours.

KEYWORDS:

Fibre cement; precuring, hydration; microstructure; XRD.

INTRODUCTION

Fibre cement is a composite material consisting of cement, silica, cellulose fibres and water. Pozzolanic materials are added to other materials to improve durability. The slurry is converted to sheets using the Hatscheck process. Excess water is eliminated through the vacuum boxes and by roller pressing. After a number of turns on the production roller the sheets are cut off onto the conveyer then transported to end of the machine to be stacked on steel form plates. These stacks are pushed through the curing chamber, at 60°C, for a minimum of 8 hours and a maximum of 72 hours, depending on the availability of autoclaves. Some production lines do not use the curing chambers. The sheets are then restacked on the autoclave carriers without the form plates. This is a critical stage where the green strength must have developed sufficiently for the lifters to be able to lift up the sheets without any damage.

When water is added to cement, several hydration reactions occur (Hewlett, 2003) and (Taylor, 1997). The calcium silicates are the only two that contribute to strength. Tricalcium silicate is responsible for most of early strength whilst dicalcium silicate, which reacts slowly, contributes only to the strength at a later stage (beyond 7 days). The reaction for the hydration of tricalcium silicate is given by the following equation:

$2C_3S + 7H \rightarrow C_3S_2H_4 + 3CH + 173.6\ kJ$ where C is CaO, S is SiO_2 and H is H_2O.

Tricalcium silicate rapidly reacts to release calcium ions, hydroxide ions and a large amount of heat. The evolution of heat is due to the breaking and formation of chemical bonds during the hydration process. The reaction continues to produce the calcium and hydroxide ions until saturation point is reached. Once this occurs, the calcium hydroxide starts to crystallize and the calcium silicate hydrate immediately forms. To ensure continuity of cement hydration, appropriate curing conditions must be applied. Some of the curing conditions used include normal curing, low pressure steam curing and high pressure steam curing. Normal curing occurs under moist and ambient temperature but the hydration process and strength development rate of cement paste

are slow (Liu, et al., 2005). Hydrothermal curing has a strong effect on mechanical properties. The major effect is the development of a denser microstructure with the formation of calcium silicate hydrate phase (Yazici, et al., 2013). It has been demonstrated that, at each autoclaving temperature, there is an optimum period of curing that results in good mechanical and physical properties (Hanson, 1963).

The goal of this paper is to use the mechanical properties data and microstructure analyses of the hydrothermally cured fibre cement sheets to study the impact of various precuring conditions.

EXPERIMENTAL MATERIALS AND METHODS

The manufacture of fibre cement sheets in this study was carried out with cement, milled silica sand, kaolin and cellulose fibres and process water. The river sand is milled with steel balls to finer particle size and the cellulose refined to a freeness of 400 – 440 Canadian Standard Freeness. Flocculant is added for dewatering purposes, retention of fine materials and settling of solids in the mud water tank.

Raw Materials

The chemical composition of the raw materials used is populated in Table 1 and physical properties of flocculant in Table 2.

Table 1: Chemical composition

Elemental oxide	Silica sand	Cement	Kaolin
Fe_2O_3 (%)	1.21	2.72	0.28
TiO_2 (%)	0.12	0.36	0.89
CaO (%)	0.07	67.36	0.01
K_2O (%)	0.53	0.42	5.38
SiO_2 (%)	91.9	21.17	67.5
Al_2O_3 (%)	0.7	4.53	24.1
MgO (%)	<0.10	1.87	0.43
Na_2O (%)	<0.10	0.06	0.38

Table 2: Physical properties of flocculant

Product Specifications	***Mesh Size (%> 10mesh): 2***
Form: Granular Solid	***Mesh Size (%< 100mesh): 6***
Colour: White	***Approx. Bulk Density: 0.80***
Ionic Character: Anionic	***Recommended working concentration (g/l): 3 - 5***
Charge Density: Medium	***Dissolution Time (min) in D.I. H_2O @5g/l at 25◦C (g/l): 60***
Molecular Weight: Very-high	

The particle size distribution curves of the above materials are graphically illustrated in Figures 1, 2 and 3 below.

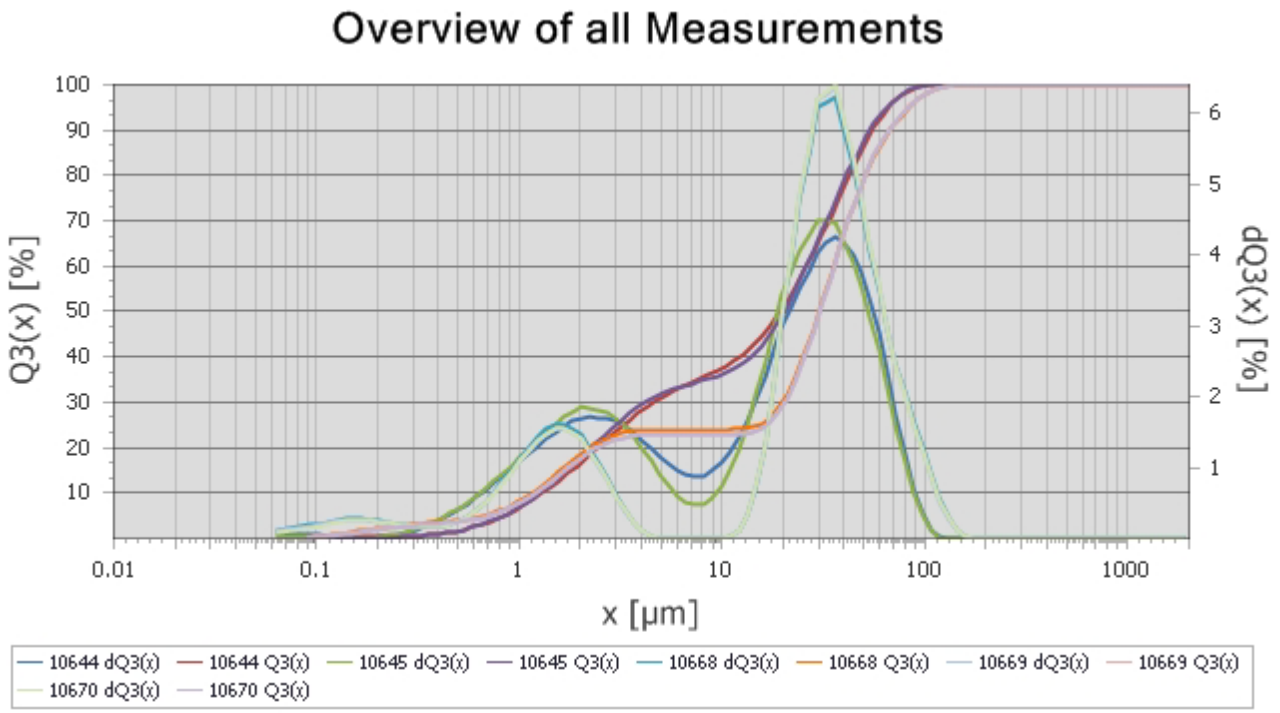


Figure 1: PSD for milled silica

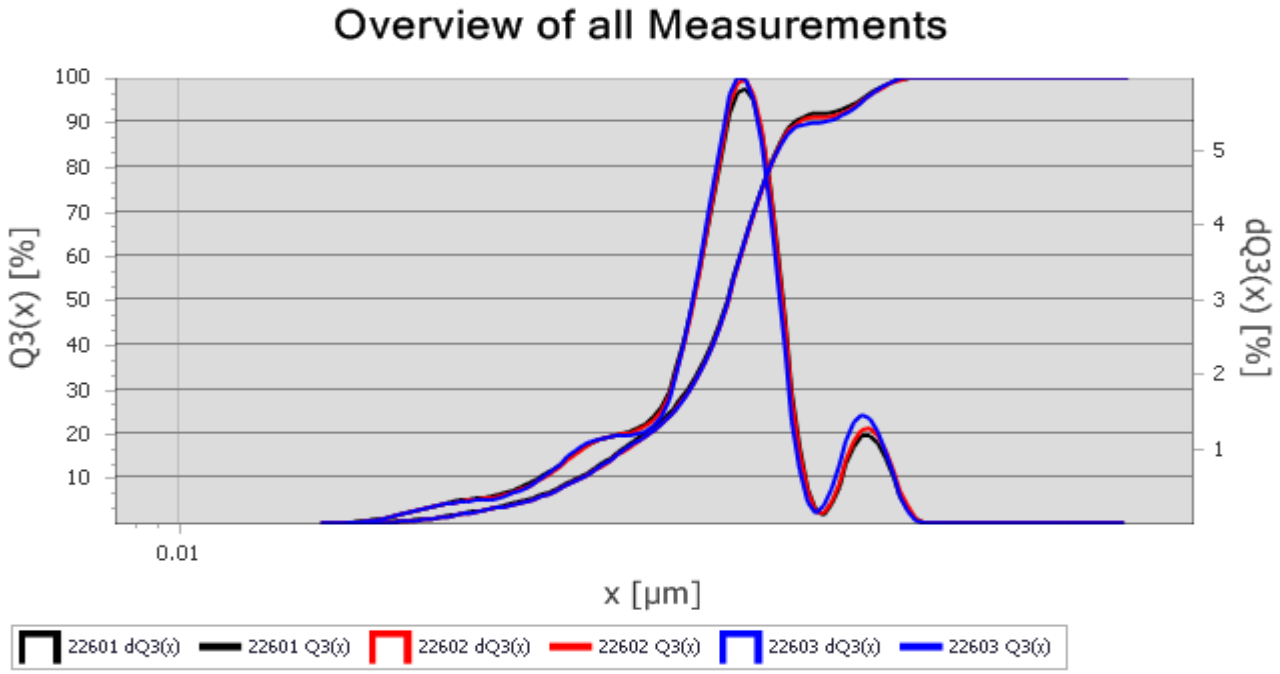


Figure 2: PSD for cement

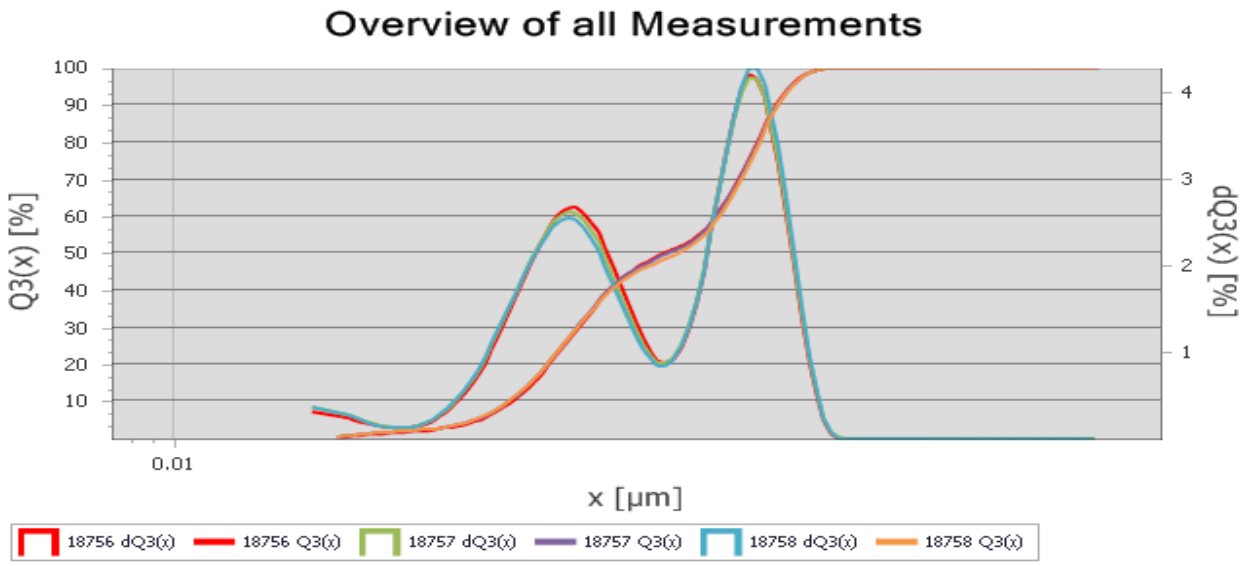


Figure 3: PSD for kaolin

Procedure

In this experiment, all the samples were prepared using the pilot Hatscheck plant. After the production roller, the sheet was cut off and placed on the conveyor where it was transported to the cutting area. Samples of 300mm x 300mm were cut and placed on steel form plates.

The pre-curing exercise, before autoclaving, was divided into five:

i. The first set of samples was cured under normal atmospheric conditions for 24 hours.
ii. The second set of samples was cured as above but the samples were shrink-wrapped with plastic.
iii. The third set was placed in a chamber at 50°C for 8 hours, shrink-wrapped in plastic, thereafter taken out of chamber and remained at atmospheric conditions for 16 hours.
iv. The fourth set was cured in the chamber for 12 hours, shrink-wrapped in plastic, thereafter at atmospheric condition for another 12 hours.
v. The last set was placed in the chamber for 24 hours, shrink-wrapped in plastic.

At the end of duration time in the chamber, the samples were unwrapped and destacked before completion of their 24 hour curing period. The samples cured under atmospheric conditions, were unwrapped and destacked after 24 hours. Thereafter, all the samples were hydrothermally cured in the autoclaves for 14 hours.

Sample Preparation and Testing

Ten specimens of each set were cut into 250mm x 250mm, dried in the oven at 80°C for 24 hours. At the end of this period, five samples of each set were tested in a Lloyd machine for mechanical tests, according to South African National Standards. The broken rectangular pieces were used for XRD and SEM analyses. XRD was done with PANalytical AERIS Diffractometer with PIXcel detector. The phases were identified using X'Pert Highscore and relative phase amounts were using the Rietveld method.

In order to determine linear expansion, the remaining five specimens of each set were taken out of the oven and cooled down to room temperature. Thereafter the length of each specimen was measured along the cellulose grain and perpendicular to the grain. The specimens were then immerse in water for 24 hours. At the end of this period, the specimens were removed from water, dabbed off excess water with a damp cloth followed by measurement of length in the direction and perpendicular to the direction of cellulose alignment in the specimen.

The linear expansion is calculated as per following equation:

$$\text{Linear expansion (mm/m)} = \frac{(L_{wet} - L_{dry}) \times 1000}{L_{dry}}$$

RESULTS AND DISCUSSION

At the end of the pre-curing period, the samples that were cured in the chamber had enough green strength and were separated with ease. For shrink-wrapped specimens pre-cured under atmospheric conditions, it was found that the specimens at the bottom of the stack were fragile and wet whereas the top and middle stacks were easy to destack. The samples that were not wrapped under the same conditions were mostly dry around the edges but the centre still wet. It was easier to remove the edges but care was required for lifting the centre. This precaution will not be possible when the process is automated. Although the specimens in the chamber were shrink-wrapped the performance was different due to the heat that accelerated the hydration reaction to form calcium hydroxide and calcium silicate hydrate crystals.

Physical properties

The density results did not show significant difference, irrespective of pre-curing conditions, Figure 4. Comparing the results to that of unwrapped specimens, the water absorption values showed a slight decrease for wrapped and heat pre-cured specimens for ≥12 hours. The decreasing trend for unwrapped, 12 hours and 24 hours pre-curing was 3.7%, 3.0% and 5.6% respectively. A significant reduction of 20% was noted in specimens pre-cured for 8 hours.

Expansion of fibre cement products is problematic for application hence it is essential that the expansion is limited to less than 1mm/m. The results in this study indicated a 17% reduction in expansion only by wrapping the specimens. Pre-curing in the chamber for 8 hours and 12 hours showed significant reduction of 27% and 33% respectively. Keeping the specimens longer in the chamber showed a 24% reduction but a 14% increase after 12 hours. Therefore, the ideal pre-curing conditions for achieving the least movement has been found to be 12 hours in the chamber wrapped and 12 hours outside unwrapped.

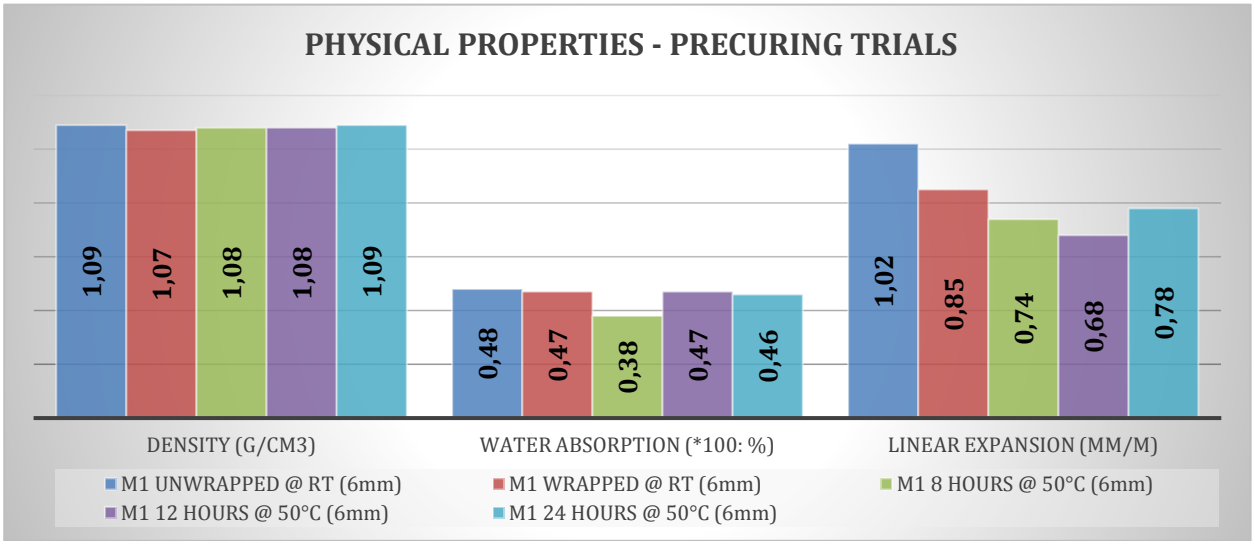


Figure 4: Physical properties of fibre cement specimens

Mechanical Properties

The flexural strengths of the pre-cured specimens are shown in Figure 5. The results presented are an average of 5 specimens. As can be observed from Figure 5, all the specimens that were pre-cured in the chamber yielded strengths lower than the unwrapped specimens pre-cured under atmospheric conditions. Although these specimens pre-cured in the chamber resulted in lower strengths, the deflection values were higher. This implied that the specimens had high fracture energy and were less brittle. It was also noted that the specimens shrink-wrapped in plastic, and pre-cured at atmospheric conditions, showed a 7% increase in strength compared to the unwrapped specimens.

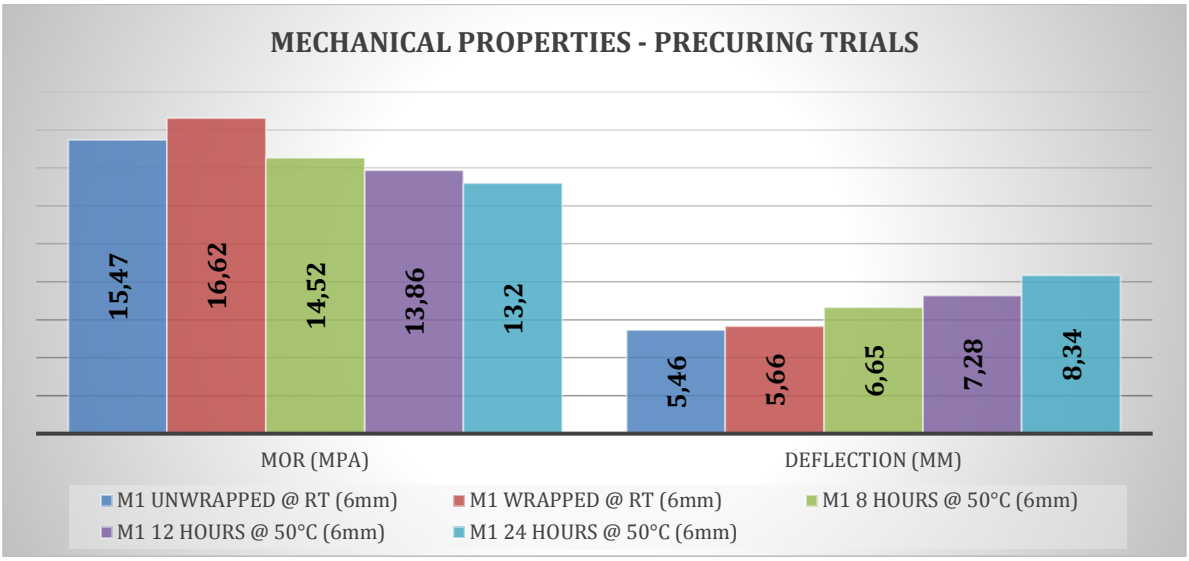


Figure 5: Mechanical properties of fibre cement specimens

Microstructure Analyses

The microstructure of autoclaved specimens was analysed using scanning electron microscope (SEM) and Quantitative X-ray diffraction (XRD). The XRD results are plotted in Figure 6 below.

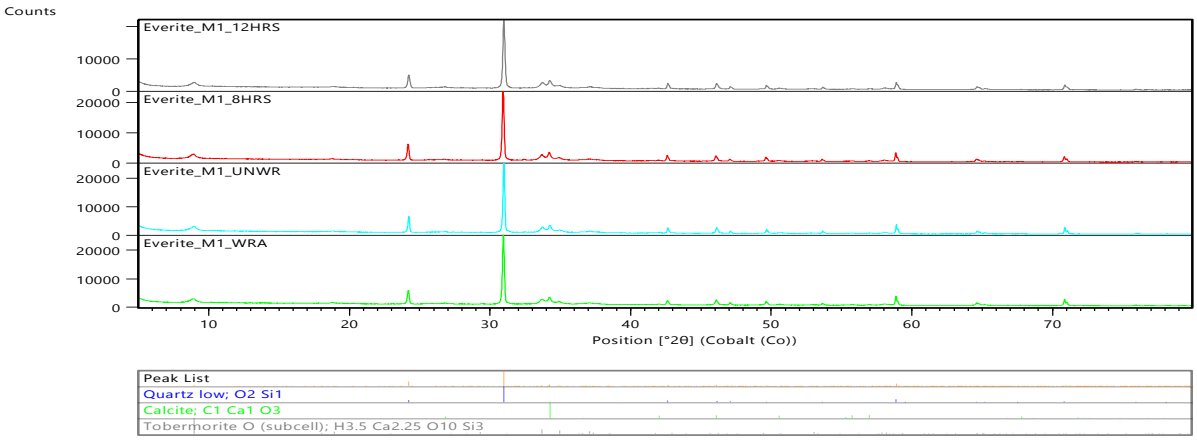


Figure 6: XRD analysis for autoclaved specimens

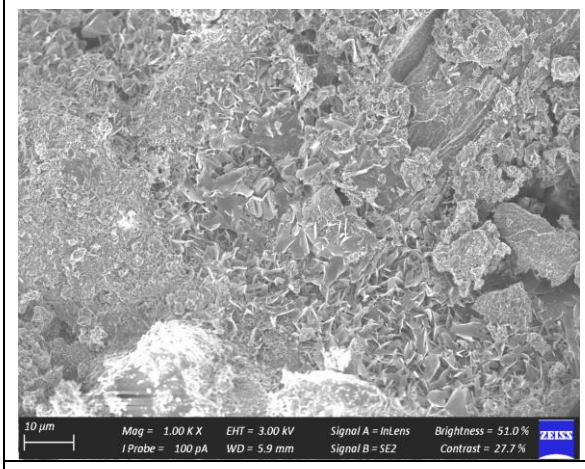
The quantitative results as per spectrum above are tabulated in Table 3.

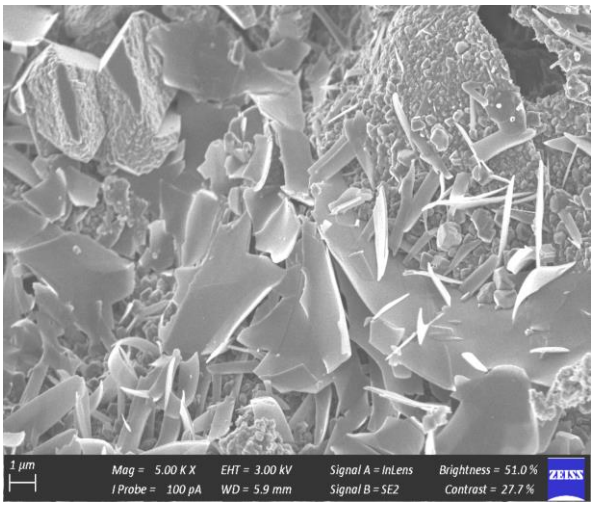
Table 3: Quantitative XRD

Specimens	Quartz	Tobermorite, 11 Å	Calcite
Unwrapped for 24 hrs. (%)	68.86	17.83	13.31
Wrapped for 24 hrs. (%)	68.72	18.9	12.39
Chamber for 8 hrs. (%)	68.76	17.84	13.4
Chamber for 12 hrs. (%)	68.49	18.04	13.47

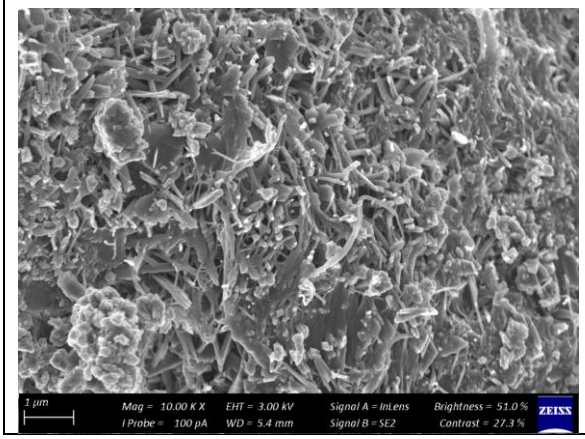
The wrapped specimens' results confirmed the strength results as the tobermorite value was higher compared to the unwrapped specimens. The tobermorite quantities for specimens pre-cured in the chamber are slightly higher than the unwrapped sample. The difference in XRD and strength results can be explained by SEM micrographs, see Figure 7.

12 hrs pre-curing in chamber





8 hrs pre-curing in chamber



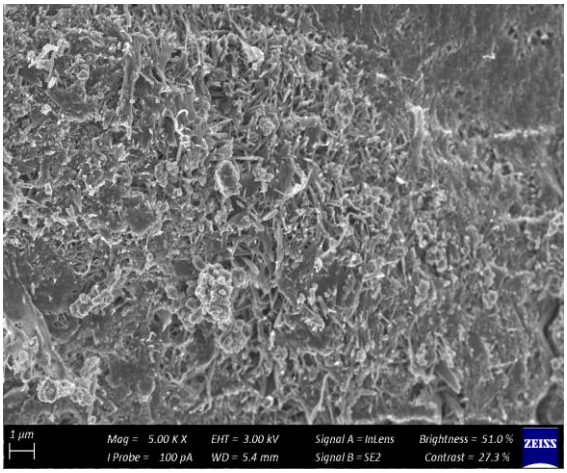


Figure 7a: SEM analyses results of specimens pre-cured in a chamber

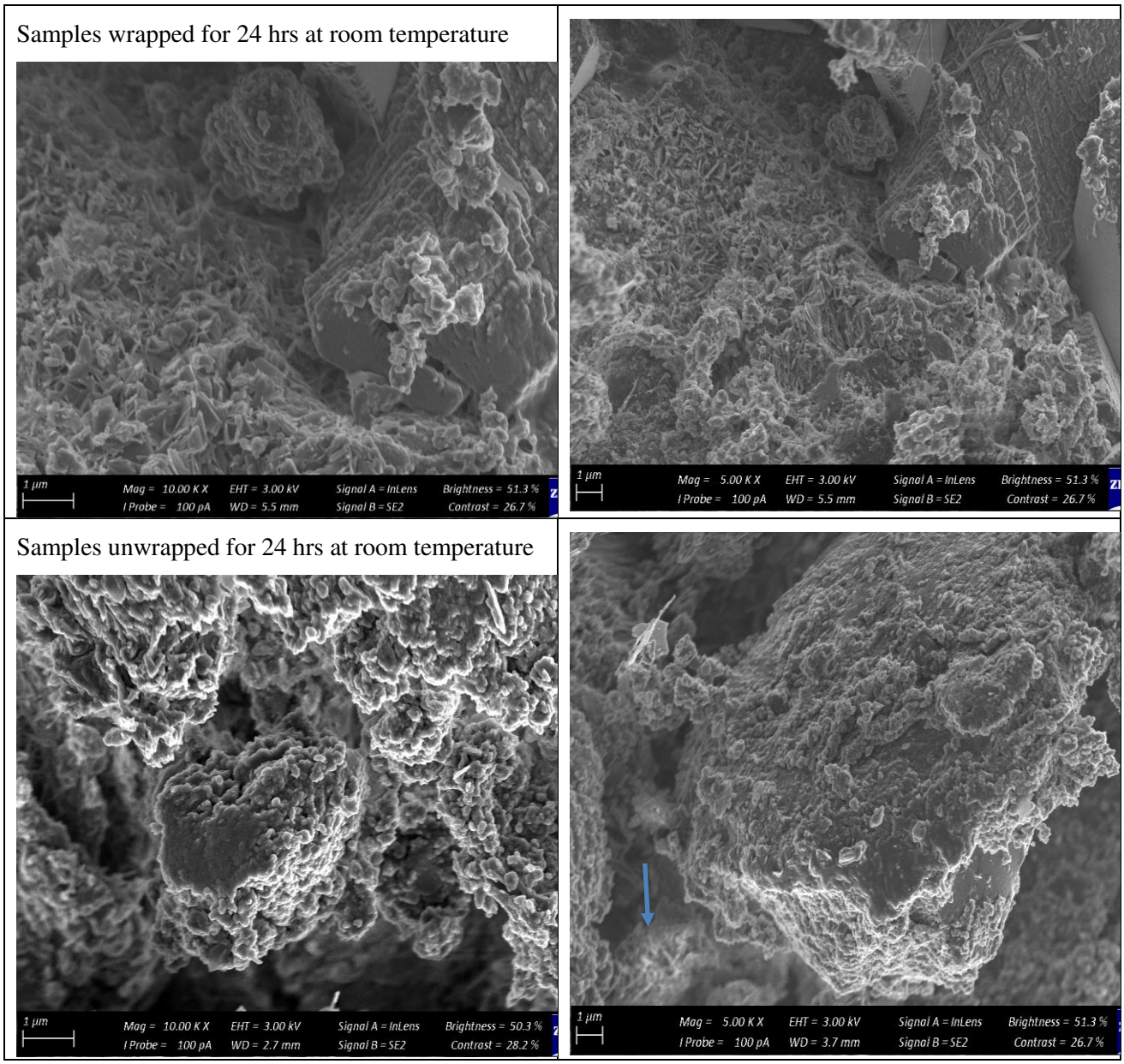


Figure 7b: SEM micrographs for atmospheric pre-curing specimens

Low strength of unwrapped specimens is attributed to the formation of fibrous-like hydrated products and loose structures, see Figure 7b. Pores were also observed. On the other hand, the wrapped specimens micrographs revealed dense, compacted and closed network structures. The enhancement in strength for these specimens could be ascribed to the fact that additional CSH phases, such as tobermorite, were formed. The formation of tobermorite filled the pores and enhanced the compactness of the hydrated mix. It has been reported that tobermorite is associated with high strength and lower permeability features (Jupe et al., 2008). The slight decrease in strength for the specimens that were pre-cured in the chamber could be attributed to the formation of some hydrogarnet (C_4SH_4) phases instead of tobermorite ($C_5S_6H_5$). The reason for the formation of hydrogarnet phases is due to the presence of Al in the system as kaolin contains a significant amount of Al_2O_3 (Table 1). It has been stated that the performance of mechanical properties of hydrogarnet is lower than that of tobermorite (Kondo et al., 1975).

CONCLUSIONS

This paper studied the effect of pre-curing, before autoclaving, on the early strength of fibre cement specimens and on the properties of autoclaved materials. The following conclusions can be deduced:

1) The SEM showed the distinct formation of crystalline platelike tobermorite and hydrogarnet in specimens that were pre-cured in the chamber. Hydrogarnet phases were more evident on the 12 hours pre-cured specimens. There were this platelike structures with broken edges observed and these crystals did not form networks hence the lower strength.
2) XRD results indicated the presence of calcite in all the samples. This is attributed to the type of cement used that contains 15% calcium carbonate. Also, the highest quantity of tobermorite phase attained was due to the wrapping of specimens under normal pre-curing conditions. These specimens showed high strength values.
3) By wrapping the specimens at the beginning of the hydration process, a less permeable matrix is formed. There are less pores in the material that are available to absorb water and expand the material. Therefore, all the shrink-wrapped specimens resulted in a reduction of linear expansion.

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