APPLICATION OF MICROSILICA IN AUTOCLAVED PRODUCTS

1. LIN ZHEN, WANG YUNANYUAN, ZHANG YI; 2. GERRY BROWN

1. Elkem Silicon Materials Fibre Cement Laboratory
Yunshan South Rd.No.17, Xidingfuzhuang, Tongzhou industry Zone, Beijing, P.R.China

lin.zhen@elkem.no

2. Elkem Silicon Materials

PO Box 8126 Vaagsbygd, NO-4675 Kristiansand, Norway
gerry.brown@elkem.no

ABSTRACT

The effect of using microsilica in air-cured fibre cement products has been investigated widely by many groups, and its benefit has been recognized by the industry. In other types of fibre cement product produced by autoclave technology, the use of microsilica has also been developing as producers move to non-asbestos products. Today, quartz sand is the traditional source of SiO_2 in autoclave recipes, so microsilica is not automatically used. However, microsilica is being used by a growing number of producers, to improve strength and durability. Microsilica is also being used in autoclaved products as a process aid and to reduce delamination in autoclaved sheets. The typical dosage of microsilica in autoclaved products to achieve significant benefit is in the range of 3% -10% of the total recipe.

This paper examines the application of microsilica in autoclaved non-asbestos flat sheets. Microsilica was tested at 3%, 5%, 7% and 10% dosage in recipes and bending strength and other physical performance was evaluated and microstructure and phases were analyzed by SEM (Scanning Electron Microscopy) and XRD (X-ray diffraction). The mechanism of the microsilica effect and the related autoclave reaction is discussed. The paper shows that microsilica can improve the bending strength, freeze-thaw resistance and other properties of autoclaved products.

KEYWORDS:

Autoclaved, Microsilica, Bending strength, Freeze-thaw resistance

INTRODUCTION

Although the autoclave process needs relatively high initial investment compared to the air-cured process it has still been chosen by many producers in the fibre cement industry for its short production time and good final product properties. Today, quartz sand is the traditional source of SiO_2 in autoclave recipes, so microsilica is not automatically used. .With the non-asbestos development in the fibre cement industry, many raw materials were tried in the new recipes, and microsilica has been used widely in the air-cured recipes in the industry. Today microsilica is also gradually being accepted by more and more producers of autoclaved products for the improvement in the property of the final product it creates.

The application of microsilica in autoclaved, non-asbestos flat sheet is introduced in this paper. 3%, 5% and 7% dosage of microsilica was tested in the recipes and bending strength and other physical performance was evaluated and microstructure and phases were analysed by SEM (scanning electron microscope) and XRD (X-ray diffraction).The mechanism of the microsilica effect and the related autoclave reaction is discussed.

EXPERIMENT

Test Materials:

Microsilica was supplied by Elkem, and the other raw materials were purchased locally

Test Procedure:

All of the raw materials were based on DRY weight in the recipe.

Sample preparation and measurement was according to Elkem Fibre cement lab standard. (Elkem FC lab standard, MAT-FC-20110105.801~806).

After sample production, all the samples were packaged in plastic bags and cured in a wooden box 50°C with a bottle of water inside for 24 hours, then each plastic bag was removed and the samples were transferred to an autoclave for curing. Autoclaving temperature and time were setup according to industry values and the autoclaving process was automatic.

After autoclaving, the samples were cured in a climate cabinet at 23±2 centigrade and 50% relative humidity for 4 days after which time the samples were ready for measurement.

CONCLUSION

1. Microsilica improved the bending strength and other physical properties of autoclaved fibre cement sheets.
2. Microsilica improved the freeze-thaw resistance of autoclaved fibre cement sheets.
3. The combination of microsilica and fillers, such as Al $(OH)_3$, improve the performance of autoclaved fibre cement products more than microsilica alone.
4. The overall contribution of microsilica in the autoclaved product was variable depending on the other raw materials. Different cellulose and fillers influence the final effect of microsilica on the final properties of the samples.
5. Both laboratory tests and industry application has shown that microsilica can improve the performance of autoclaved products.

RESULTS AND DISCUSSION

The effect of microsilica on the primary physical properties of autoclaved sheets was evaluated. Different types of cellulose were used in the test and 7% Microsilica was evaluated.

The following properties of the samples were evaluated in the test.

Bending strength

Dry density

Water absorption

Freeze-thaw resistance

Effect of microsilica dispersion efficiency

Test formulations are listed in Table1 and test results are shown in figure 1 to figure 4.

Table 1 - Test formulations

Sample ID	Cement (P.O. 42.5)	Microsilica	Quartz sand (7500 bl.)	Pulp(NUKP , 51°, kcl)	Pulp(PM, 25-30°SR)	Pulp(Tasmann)	Pulp(Kinlith)
PMA	45%	0%	45%	10%	0%	0%	0%
PMB	45%	0%	45%	0%	10%	0%	0%
PMI	45%	0%	45%	0%	0%	10%	0%
PMJ	45%	0%	45%	0%	0%	0%	10%
PMD	45%	7%	38%	10%	0%	0%	0%
PMC	45%	7%	38%	0%	10%	0%	0%
PMK	45%	7%	38%	0%	0%	10%	0%
PML	45%	7%	38%	0%	0%	0%	10%

Bending Strength

Bending strength results are shown in figure1.Figure1 shows that bending strength was increased as microsilica was added. The increase was different based on the cellulose. In this test, bending strength was increased 0.5MPa to 3.6MPawhen 7% microsilica was used, an increase level of 3% to 46%compared to the reference. It was shown that the microsilica had a beneficial effect on the bending strength of the autoclaved fibre cement sheet and this effect was influenced by the type and quality of the cellulose.

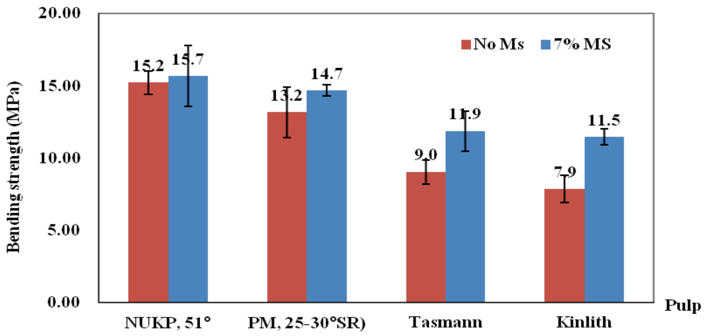

Figure1. Bending strength (MPa)

Dry Density

Dry density results are shown in figure2. It is shown in figure2 that dry density also increased as microsilica was used,, increasing between 0.03g/cm3and 0.14g/cm3depending on different cellulose, an increase level of 3% to 13% compared to the reference. Dry density generally changed in line with the bending strength, which means high strength equates to higher density.

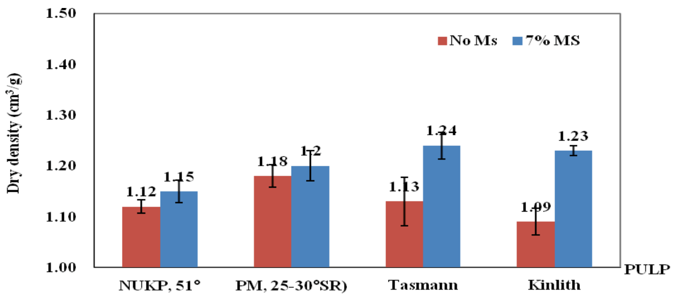

Figure2. Dry density

Water Absorption

Water absorption results are shown in figure 3. It is shown that water absorption decreases as microsilica was used in the test, decreasing 2% to 8% as 7% microsilica was used. Water absorption change is in line with the dry density, which means higher water absorption equates to lower density.

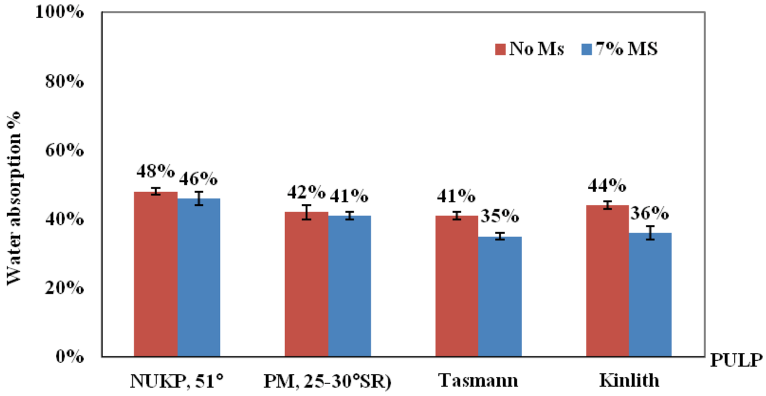

Figure 3 . Water absorption

Freeze-thaw resistance

The effect of microsilica on freeze-thaw resistance was studied. 3% dosage of Microsilica was used in the recipe, and for reference, another filler $Al(OH)_3$ was also studied according to the requirement from the producer. Test formulations are listed in table2 and test results are shown in figure 4 to figure 5. Figure 4 shows the bending strength remaining after 100freeze-thaw cycles (-20C to +20C).Figure 5 shows the appearance of the sample after the freeze-thaw test.

Table 2 - Test formulation

Sample ID	Cement (P.O325)	Microsilica	Al(OH)3	Bleached Pulp	Quartz sand
ASREF	42 %	0.0 %	0.0 %	8.0 %	50.0 %
AS1	42 %	0.0 %	3.0 %	8.0 %	47.0 %
AS2	42 %	3.0 %	0.0 %	8.0 %	47.0 %
AS3	42 %	1.5 %	1.5 %	8.0 %	47.0 %

It is shown in figure 4 that the sample AS2 with 3% Microsilica had the highest strength of all the samples at 11.0MPa.This was decreased to 9.1MPa after 100 freeze-thaw cycles. The strength remaining was 83% compared to the reference sample. The reference sample without microsilica or $Al(OH)_3$had 5.9MPa strength remaining after the test which was 61% of the reference. The sample with 3% $Al(OH)_3$had 5.1MPa remaining which was58% compared to the reference. The sample with a combination of 1.5% microsilica and 1.5% $Al(OH)_3$ had 7.7MPa strength remaining which was 83% of the reference .

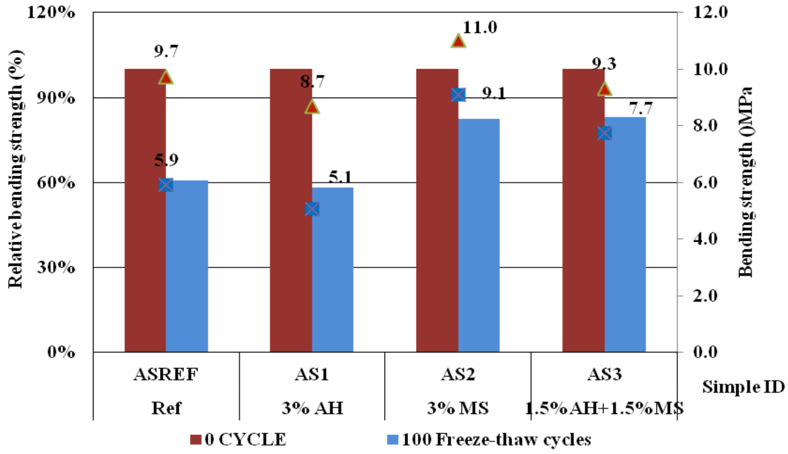

Figure 4. Remaining bending strength after 100 F/T recycles.

Figure 5 shows the appearance of samples after 100 freeze-thaw cycles, itis shown that the sample AS2 and AS3 with microsilica had no visible cracks on the surface,but all other samples did show cracks on the surface.The reference samplewas the worst of all.

The freeze-thaw testing proves that microsilica significantly improves freeze-thaw resistance.

Figure 5 - Appearance of sample after 100 freeze-thaw cycles

Effect of dispersion efficiency ofmicrosilica

It has been proved that microsilica improves the properties of autoclaved fibre cement products, particularly the durability. The contribution of microsilica in the product is affected by several factors and the dispersion efficiency of microsilica is the key influence on the final contribution of microsilica in the final product. We studied the effect of dispersion efficiency of microsilica on the autoclaved fibre cement product. Sample production was the same as the previous test, and microsilica was added into the process with two options,

a) Microsilica was added into fibre cement recipes as pre-made slurry with good dispersion efficiency.

b) Microsilica was added into the fibre cement recipe as the last component as dry powder, which is known to give poor dispersion efficiency, requiring increased mixing time and mixing energy to achieve proper dispersion.

10% of microsilica was tested in the recipe, and six different microsilica recipes were tested. Six different microsilica samples were produced at different times at the same plant, so their chemical composition was slightly different. SiO_2 content of the six microsilica samples is shown in the figure 6.Bending strength is shown in figure 6 too.

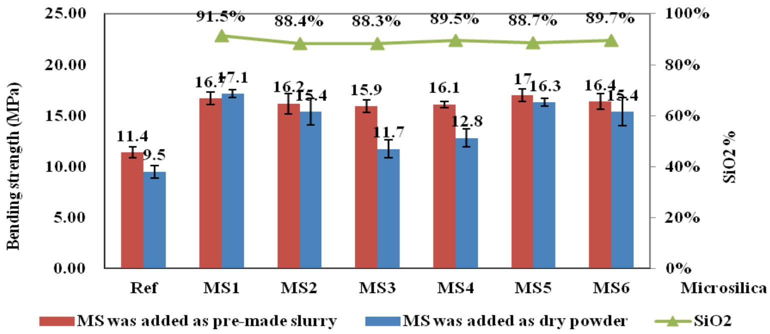

Figure 6 Bending strength of the sample as Microsilica was added as pre-made slurry and dry powder

It is shown in figure 6 that all the samples showed increased bending strength as microsilica was added. Apart from sample Ms1, the other samples showed higher strength as microsilica was added as pre-made slurry compared to the recipes using dry microsilica powder directly.

SiO_2 is normally the main active element in microsilica. Figure 6 shows that higher SiO_2level does not always produce higher strength in autoclaved products. SiO_2 percentage of the six samples varied from 88.3% to 91.5% a range of~3%.

To better understand the effect of dispersion efficiency of microsilica on its final level of effect, we made another test to evaluate the difference in effect as microsilica was used in dry powder form and as a pre-made slurry. Three types of microsilica that were produced at different times, MS-A, MS-B and MS-C were tested. Each sample was added into the process as pre-made slurry and dry powder. Figure 7 shows the strength results when the microsilica was added as pre-made slurry; and figure 8 shows the strength result when microsilica was added as dry powder. It is shown in figure 7 that strength was increased ~40%when 10% Microsilicawas added as pre-made slurry, and variation of this effect was small between the samples in the slurry group. For the other group of samples made with dry microsilica powder, figure 8 shows that strength also increased but the spread of the results was very high. It was proven that good dispersion quality of Microsilica was necessary and important for consistent final fibre cement product.

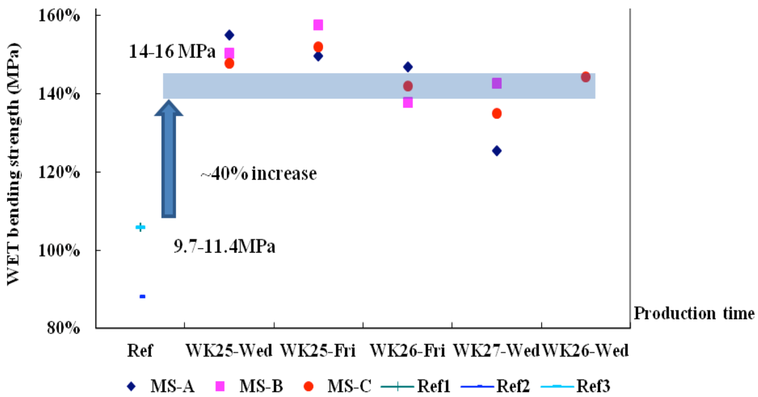

Fig 7 Microsilica was used as pre-made slurry

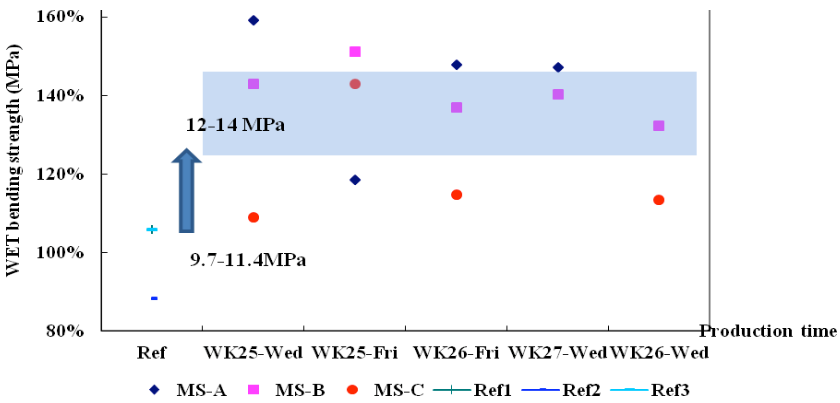

Fig 8 Microsilica was used as dry powder after cement

DISCUSSION

It was confirmed from above results that microsilica is beneficial in improving the properties of autoclaved fibre cement products. For autoclaved fibre cement product, ~40% quartz sand was used in the recipes, and the main ingredient, crystalline SiO_2 in the form of sand was not able to fully react with lime at room temperature.

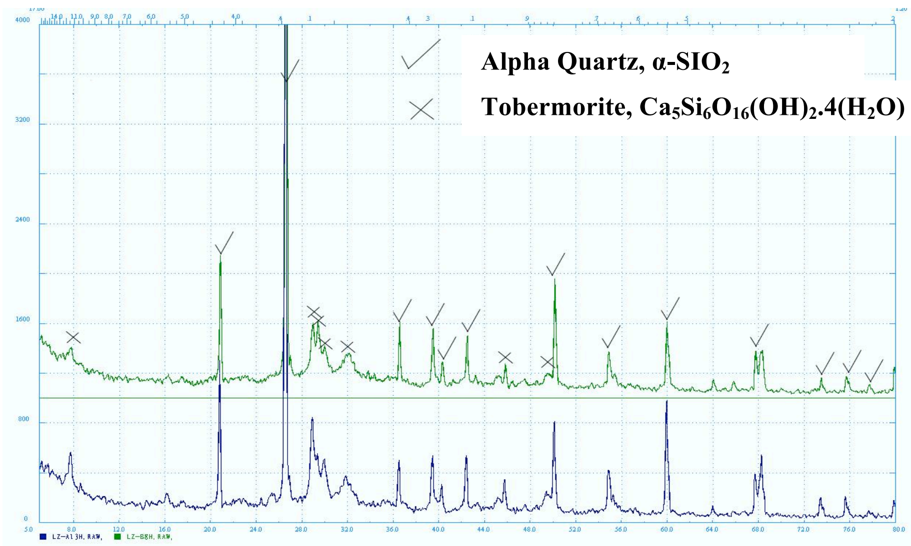

Fig 9 - XRD pattern of Autoclaved fibre cement

Even under autoclave conditions, quartz sand is not able to react totally in practice, and there is always some left. We know that the autoclave product Tobermorite ($Ca_5Si_6O_{16}(OH)_2.4(H_2O)$ or Xonotlite($Ca_6Si_6O_{17}(OH)_2$),created by the reaction between $Ca(OH)_2$ and SiO_2under autoclaving condition will give visible contribution to the final strength of the autoclaved product. Insufficient reaction due to poor autoclaving or poor recipes would lead to poor strength of the final product. Some producers had to increase the autoclaving temperature or prolong the autoclave time to guarantee enough reaction occurs. Figure 9is an XRD pattern to show that a longer time in the autoclave would produce more Tobermorite in the autoclaved fibre cement product. However increasing autoclaving time is an energy consuming process, Microsilica is able tore act with $Ca(OH)_2$to create more binder at room temperature due to its high Pozzlanic characteristics, which results in the strength improvement. Theoretically, Microsilica is also helpful in the formation of Tobermorite because more binder (CSH gel) is formed at lower temperature, and CSH gel will be transformed into Tobermorite at autoclaving temperatures. Industry application and laboratory test results have shown the visible strength reinforcement in the autoclaved fibre cement product when microsilica is used.

ACKNOWLEDGEMENTS

The author would like to thank the following peoples for supporting this study and creating the paper.

Gerry. Brown E-mail: gerry.brown@elkem.no **Magne.dastol** E-mail: magne.dastol@elkem.no **Zhang Yi** E-mail: zhang.yi@elkem.no **Melissa Wang** E-mail: melissa.wang@elkem.com	**Simon. Wison** E-mail: simon.wilson@elkem.no **Roy. Saptak** E-mail: saptak.roy@elkem.no **Hironori.Yoshizawa** E-mail: hironori.yoshizawa@elkem.no **Surachai Suttitumma** E-mail: surachai.suttitumma@elkem.com

REFERENCES

[6]. Dr. ShenRongxi, New type fibre reinforced cement-based composite, ISBN 7-80159-609-9, 2004, China

[7]. Dr. Benoit de Lhoneux, the microstructure and ageing of cellulose fibre reinforced autoclaved fibre cement composites. International Journal of Cement Composites and Lightweight Concrete, Volume 11, Issue 2, May 1989.

[8]. Internal report, Elkem Fibre cement lab.

[9]. Lin Zhen, Zhang Yi, Effect of ElkemMicrosilica on the Air-Cured Asbestos-Free Fibre Cement Product, the 6th International Symposium on Cement & Concrete, Xi'an, P.R.China , September 19~22 2006

[10]. Schreiner & Holmen, The effect of Microsilica dispersion in fibre cement mixes, 3rd international inorganic-bonded fibre composites conference, 2002

[11]. T M Cooke, the effect of aluminous Additives on the properties of Autoclaved cellulose fibre cement,11th international inorganic-bonded fibre composites conference, 2010