

SUPERIMPOSED REINFORCEMENT EFFECT OF MICROSILICA AND FIBRES ON FIBRE CEMENT PRODUCT

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ABSTRACT

Wood pulp and PVA fibre have been widely used in fibre cement products since the asbestos fibre was banned. PULP or PVA fibre normally does not bond well with the cement matrix due to its inherent difference from the cement hydration phase, therefore the interfacial zone between the fibre and cement hydration phase would be the weak part in the matrix. Reinforcement of the interface bonding strength will improve the contribution of the fibres. Moreover, as a natural fibre, PULP has the risk of degradation over time; prolonging the service life of PULP is valuable work for the fibre cement product. Microsilica is one of the additives widely used in nonasbestos fibre cement products; besides its reinforcement function, which is attributed to its pozzolanic character, it also serves as an excellent medium to enhance the interfacial property between fibre and cement hydration phases. In this study, the aim is to understand more about the superimposed reinforcement effect of Microsilica and fibres on the fibre cement product with the purpose of optimizing the use of fibres. In particular, characteristics such as physical-mechanical properties of PULP and PVA fibres, durability of the PULP, and interfacial adhesion engineered to improve the performance of the composite materials were discussed in this paper.

KEYWORDS:

Fibre cement, Microsilica, PVA fibre, Pulp

INTRODUCTION

The production of non-asbestos fibre cement products in China has developed rapidly over the past 20 years (Shen and Lin, 2006) or (Zeng and Fu and Zheng, 2016) or (Lin and Zhang and Li, 2016). As the primary substitute fibre for asbestos, the production and application of PVA fibre have been developing well. In addition to the essential raw materials such as cement and fibres, many auxiliary materials and chemical additives were also used. Microsilica was one of the auxiliary materials that was accepted by the industry and widely used for its excellent reinforcement effect, particularly in formulations using PVA fibre, Microsilica was a highly recommended material. This was not only based on the reinforcement effect of Microsilica on fibre cement products but also on the fact that its presence effectively improves the bonding performance between fibres and the cement matrix, which significantly enhances the contribution of fibre reinforcement.

EXPERIMENT.

Test materials:

The tested raw materials included cement, PVA fibre, and wood pulp, which were common industry products available in the market. Microsilica was supplied by Elkem. Specification of the raw materials and additives are listed in the appendix.

Test procedure:

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

The preparation and measurement of the fibre cement samples were conducted in accordance with Elkem's Fibre Cement Lab Standard.

After the samples were prepared, they were placed into sealed plastic bags and then into a wooden box containing a bottle of water at 50°C for pre-curing for 24 hours. Subsequently, the samples were transferred to a drying cabinet at 50°C for 6 days of curing, and then removed from the sealed plastic bags and moved to a climate cabinet maintained at 23±2°C and 50% relative humidity for an additional 4 days of curing. Afterward, the samples were ready for measurement.

Test 1 primary effect of Microsilica on the fibre cement product

Depending on the curing systems, the raw materials for producing fibre cement products are generally divided into two categories. One category is based on the air-cured curing system and mainly consists of cement and fibres and some auxiliary materials. PVA fibre can only be used in this air-cured fibre cement recipe, with the basic strength and main mechanical properties of this fibre cement product primarily derived from cement hydration binders. Some auxiliary materials are also used in the recipe for various purposes; some materials increase the strength as reinforcement elements, while others serve as process aids.

One test to evaluate the reinforcement effect of Microsilica on the local raw materials from one fibre cement factory was made in Elkem fibre cement technical centre. The dispersion of Microsilica directly affects its reinforcement effect (Schreiner and Holmen, 2002) or (Lin and Zhang and Wang, 2012). To avoid any impact on test results due to variations in dispersion quality, all Microsilica samples were pre-made slurry in the experiment. After ensuring that the dispersion quality was satisfactory, they were added to the sample preparation process according to the specified dosage. Test formulation was listed in Table 1.

Sample ID	∠ement	Pulp \lceil	Microsilica	PVA 1# fibre	Filler	Total
$Ref-1$	76.20%	6.00%	0.00%	$.80\%$	16.00%	100.00%
Test MS-1	72.20%	6.00%	4.00%	$.80\%$	16.00%	100.00%
Test MS-2	68.20%	6.00%	8.00%	$.80\%$	16.00%	100.00%

Table 1 Reinforcement effect of Microsilica on fibre cement

Bending strength was shown in Figure 1. As 4% Microsilica was used, the WET bending strength of the sample

increased to 10.4 MPa from the reference 9.0 MPa, an increase of 16%; when 8% Microsilica was used, the WET bending strength increased to 12.1 MPa from the reference 9.0 MPa, an increase of 34%. It was proven that Microsilica could significantly improve the bending strength.

The excellent reinforcement contribution of Microsilica was attributed to its high pozzolanic activity. Pozzolanic activity means that the active SiO2 in Microsilica reacts with Ca(OH)2 produced by cement hydration, creating more C-S-H binder and resulting in a denser matrix structure. Consequently, the final strength of fibre cement was increased accordingly. Elkem has studied the different auxiliary materials in fibre cement, and Microsilica was proven to be the best reinforcement material among the common auxiliary materials (Lin and Zhang and Li, 2018).

Test 2 the main function of PVA fibre in the fibre cement product

PVA fibre was the critical fibre for non-asbestos air-cured fibre cement products. There are also many studies on the reinforcement of PVA fibre (Hideki et al, 2008) or (Zhang et al, 2016). Three types of PVA fibres commonly used in the fibre cement industry were tested in the Elkem lab; test formulations are listed in Table 2.

Sample ID	ement	Pulp	Microsilica	$1#$ PVA	$2#$ PVA	$3#$ PVA	Filler	Total
Reference	81.5%	3.5%	5.0%	0.0%	0.0%	0.0%	10.0%	100.0%
$1#$ PVA	80.0%	3.5%	5.0%	$.5\%$	0.0%	0.0%	10.0%	100.0%
2# PVA	80.0%	3.5%	5.0%	0.0%	$.5\%$	0.0%	10.0%	100.0%
$3#$ PVA	80.0%	3.5%	5.0%	0.0%	0.0%	$.5\%$	10.0%	100.0%

Table 2 Reinforcement effect of PVA fibre on fibre cement

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The bending strength of the samples is shown in Figure 2. It was indicated from this test that the WET bending

strength of the samples containing 1.5% PVA fibre was all increased compared to the reference sample, increasing to 11.0 MPa, 11.3 MPa, and 12.9 MPa from 7.6 MPa of the reference sample, an increase of 45%, 49%, and 69% respectively, depending on the different PVA fibres.

In addition to reinforcing the strength, PVA fibres significantly impact the toughness of fibre cement products. When fibre cement samples were tested for bending strength, the area under the load-deflection curve reflects the toughness performance of the fibre cement products, i.e., the bending work. The higher the bending work, the higher the product's toughness (Lin and Zhang and Wang, 2008) or (Lin and Zhang and Wang, 2010). Bending work results of the samples were shown in Figure 3. It was shown that the bending work of the sample was significantly improved with the use of PVA fibre. The reference sample without PVA fibres exhibited a very low bending work, despite containing 3.5% wood pulp fibre. Compared with the reference sample, the bending work of the samples added with different PVA fibres increased from 66 N·mm of the reference sample to 774 N·mm, 784 N·mm, and 1197 N·mm. This result proves the importance of fibres in fibre cement products. Especially when both wood pulp fibre and PVA fibres were present in the product, the PVA fibres were the key factor determining the product's toughness, rather than wood pulp fibre.

Test 3 synergistic effect of using Microsilica and PVA fibre

Even if the fibres themselves possess excellent properties, such as high strength and high elastic modulus, if the bond strength between the fibres and the cement matrix is poor, then these fibres cannot effectively enhance the strength. Therefore, improving the bonding strength between the fibres and the cement matrix is a crucial measure to ensure that the fibres can effectively function in the fibre-cement matrix. When the fibres' effective contribution is enhanced, the overall strength and toughness of the product are correspondingly improved. matrix.

Elkem Fibre Cement Technical Centre has studied the synergistic effect of Microsilica and PVA fibre on the non-asbestos air-cured fibre-cement product, focusing on the bending strength and toughness. Different dosage combinations of Microsilica, PVA fibre, and wood pulp were included in the formulation. One type of PVA

fibre, one type of wood pulp, and two different Microsilica samples were used in this experiment.

The specific experimental formulations were listed in Table 3. The test results of bending strength and bending work were shown in Figures 4 to Figure 7.

Sample ID	Cement	Pulp	Microsilica 1	Microsilica 2		total
			$(MS-PD)$	$(MS-YD)$	PVA 4# fibre	
Reference	96.50%	3.50%	0.00%	$ 0.00\%$	0.00%	100.00%
Sample-3 (MS-PD)	88.00%	4.00%	7.00%	0.00%	1.00%	100.00%
Sample-4 (MS-PD)	88.50%	3.00%	7.00%	$ 0.00\%$	1.50%	100.00%
Sample-5 (MS-PD)	85.00%	3.00%	10.00%	$ 0.00\%$	2.00%	100.00%
Sample-6 (MS-PD)	88.00%	3.50%	7.00%	0.00%	1.50%	100.00%
Sample-8 (MS-PD)	90.00%	4.00%	4.00%	$ 0.00\%$	2.00%	100.00%
Sample-9 (MS-PD)	87.50%	3.50%	7.00%	0.00%	2.00%	100.00%
Sample-3 (MS-YD)	88.00%	4.00%	0.00%	7.00%	1.00%	100.00%
Sample-4 (MS-YD)	88.50%	3.00%	0.00%	7.00%	1.50%	100.00%
Sample-5 (MS-YD)	85.00%	3.00%	0.00%	10.00%	2.00%	100.00%
Sample-6 (MS-YD)	88.00%	3.50%	0.00%	7.00%	1.50%	100.00%
Sample-8 (MS-YD)	90.00%	4.00%	0.00%	4.00%	2.00%	100.00%
Sample-9 (MS-YD)	87.50%	3.50%	0.00%	7.00%	2.00%	100.00%

Table 3 Synergistic effect of Microsilica and fibres

Figure 4 illustrates the strength of samples with varying Microsilica dosages and a constant 2% PVA fibre content. In this series of tests, the experimental results obtained with MS-PD and MS-YD Microsilica exhibited no significant difference. It was indicated from Figure 4 that the bending strength of the samples in the group utilizing Microsilica and PVA fibre increased significantly compared to the reference sample. Moreover, the results indicate that with an increase in Microsilica content, the material's strength tends to further improve. Taking the experimental group using MS-YD Microsilica as an example, when the Microsilica content increased from 4% to 7% and then to 10%, the sample's bending strength increased from 6.7 MPa of reference sample to

11.2 MPa, 12.1 MPa and 13.2 MPa subsequently. It was shown that the formulation containing the highest

Microsilica component achieved the highest bending strength when the PVA dosage was constant. Furthermore, the results in Figure 4 demonstrate that in the system where wood pulp fibre, PVA fibre, and Microsilica were used in combination, both PVA fibre and Microsilica had a significant impact on the sample bending strength, while the impact of wood pulp fibre was considerably weaker.

Figure 5 illustrates the strength of samples with different fibre contents and constant Microsilica content of 7%. According to the results in Figure 5, irrespective of whether MS-PD or MS-YD Microsilica was used, the bending strength of samples with different fibre compositions does not exhibit significant differences when the Microsilica content remains constant at 7%. All values fall within the range of 11.3 to 12.3 MPa.

Based on the above results, it was indicated that reinforcement effect of Microsilica was more effective than PVA fibre on the bending strength, Additionally, 3%-4% wood pulp did not significantly impact the strength of the samples when Microsilica and PVA fibre were present.

Figure 6 illustrates the bending work of samples with different fibre contents and a constant Microsilica content of 7%. It was indicated from Figure 6 that the bending work of both groups of samples increases notably with the rise in total fibre content, regardless of the type of Microsilica used. For the MS-YD experimental group,

Sample-9, with the highest total fibre content of 5.5% (2% PVA + 3.5% pulp), achieved the highest bending

work of 1146 N.mm; for the MS-PD experimental group, Sample-9, with the highest total fibre content also reached the highest bending work value of 1832 N.mm, which was also the highest among all experimental samples.

Comparing samples-3 and sample-6, both with the same total fibre content of 5%, sample-6, which has a higher PVA fibre content, exhibits a higher bending work. Moreover, as the PVA fibre content increases, the bending work also continues to rise. This indicates that PVA fibres have a more pronounced effect on the material's toughness than wood pulp.

Figure 7 presents the bending work of samples with different Microsilica contents and a constant 2% PVA fibre content. Generally, an increase in fibre content improves the material's toughness, but in this group of experiments, the sample with the highest total fibre content was sample-8 with 6% total fibre volume content, yet its bending work value was not the highest. For the MS-YD experimental group, sample-9 with a total fibre volume content of 5.5% (2% PVA + 3.5% pulp) and a Microsilica content of 7% achieved the highest bending work of 1832 N.mm; for the MS-PD experimental group, sample-5 with a total fibre volume content of 5% (2% PVA + 3% pulp) and 10% Microsilica achieved the highest bending work value of 1622 N.mm. This indicates that, in addition to the role of the fibres themselves, the role of Microsilica also affects the material's toughness, but the impact of Microsilica on the material's toughness was not as significant as that of PVA fibres, and its impact was mainly achieved by affecting the interfacial properties between the fibres and the substrate.

Figure 8 was a SEM image of the morphology of hydration products on the surface of PVA fibres in the fibre

cement experimental sample. It was clearly visible that there were more and denser hydration products on the PVA fibre surface when Microsilica was added. This indicates that Microsilica can effectively increase the amount of hydration products between PVA fibres and the cement matrix, resulting in a denser interfacial region and effectively improving the bond strength between the fibres and the matrix. Thus, the effective contribution rate of the fibres was enhanced, and the toughness of the material was strengthened.

Reference sample Containing Microsilica sample

Figure 8 Cement hydrated product on the PVA fibre

WOOD PULP FIBRES IN THE FIBRE CEMENT

Wood pulp fibre in the production of non-asbestos fibre cement products was primarily used to pick up various powdery materials within the raw materials, ensuring the stable production process. For the air-cured nonasbestos fibre cement products, due to the presence of PVA fibres, the usage of wood pulp fibre was generally only 3-4%. Its effect on the strength and toughness of the product was much less than that of PVA fibres, as confirmed by the aforementioned experiments. However, for the autoclaved non-asbestos fibre cement products, since only wood pulp was used in the formulation due to PVA fibre cannot withstand the high temperature and high pressure hydrothermal environment of autoclave curing, and wood pulp fibre was not only used as a process fibre, but also as a reinforcing fibre to the autoclaved fibre cement product, its typical dosage was 8-10%. Durability of the PVA fibre was excellent in the air-cured fibre cement product (Masakazu and Yamamoto and Hideki, 2006). However as a natural fibre, wood pulp was naturally degradable, its degradation was influenced by the environment. The high alkalinity environment of cement products and the extreme high temperature and pressure conditions of autoclave systems, which were harsher than those found in the natural environment. Understanding the wood pulp fibres durability and effect factors within fibre cement products was crucial (De Lhoneux et al, 2008) or (Charles and John, 2003) or (Gustavo, 2014). We conducted preliminary research and analysis on the properties of two commonly used wood pulp samples in the local fibre cement factory...

Test 4 Durability of the wood pulp in the fibre cement.

Two common unbleached pulp samples were offered by the local fibre cement factory.

Pulp 1 sample was used to study the effect of the alkalinity environment on the pulp.

Pulp 2 sample was used to study the effect of the autoclaving environment on the pulp.

The properties of the pulps were mainly determined by three main components: lignin, hemicellulose, and cellulose. The main functions of these three components in pulp fibre can be summarized as follows. Lignin in and support function. Generally speaking, the higher the lignin content, the stiffer the fibre. Hemicellulose was similar to the aggregate in concrete materials, playing a role of material supplementation and bridging. Cellulose was similar to the steel bars in concrete, mainly enhancing toughness. The single fibre strength of the pulp was positively correlated with the degree of polymerization (DP) and intrinsic viscosity index.

Cement hydration was a heat-releasing process, and the autoclaving environment was even a high-temperature and high-pressure environment.

Based on pulp characters and fibre cement curing characteristics, we measured the following parameters of the wood pulp as indicators to evaluate the property of the pulp fibre in the fibre cement. The three main components of lignin, hemicellulose, and cellulose. Degree of polymerization (DP) and intrinsic viscosity index. Wood fibre thermal stabilization determined by TG/DTG analysis. The sample preparation and treatment process are presented in Table 4.

Table 4 Sample preparation for the test of alkalinity effect on the pulp

Sample 1# and Sample 2# were treated from Pulp 1, which were used to study the effect of an alkaline environment on the wood pulp.

autoclaving environment on the wood pulp.

Figure 9 illustrates the variation in the three primary components of lignin, cellulose, and hemicellulose for the two Pulp 1 samples, compared to the reference sample 1#, the alkaline sample 2# indicates that all three main components have decreased: the total lignin content dropped to 2.83% from 3.77%; the hemicellulose content decreased to 4.47% from 5.81%; and the cellulose content decreased to 81.13% from 83.99%. This demonstrates that the pulp degrading in an alkaline environment.

Figure 10. TG/DTG chart of the two pulp 1 samples.

The TG/DTG diagram is presented in Figure 10. The decomposition temperature of the reference sample 1# was 363.45°C, which was higher than the 338.25°C of the alkaline sample 2#. The weight loss of the alkaline sample 2# was 64.1%, which was greater than that of the reference sample 1# at 62.21%. This indicates that the thermal stability of wood pulp fibres decreases in an alkaline environment.

The degree of polymerization (DP value) and intrinsic viscosity index are listed in Table 5. It is shown that the DP value of the alkaline sample 2# was 379, which was slightly higher than the 337 of the reference sample 1#. This is mainly because the alkaline solution caused a partial dissolution of the small molecular parts within the

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fibre, i.e., lignin and hemicellulose. The primary contribution to the DP value comes from the long-chain

structure of high molecular weight. When the short-chain and small molecular parts of the hemicellulose dissolve, the total mass of the substance decreases, resulting in a smaller denominator, which in turn causes the calculated DP value to increase.

Table 5 Degree of polymerization (DP value) and Intrinsic viscosity index

For the autoclaved pulp samples, compared with the original state of sample 3#, the degree of polymerization of samples 4# and 5# decreased significantly after autoclaving. The DP value of sample 4# decreased to 235 from the initial state's 1277 of reference sample 3#, and that of sample 5# decreased to 239. The results indicated that, regardless of whether the environment was alkaline or neutral, the autoclaving environment led to a significant decrease in the degree of polymerization of the pulp. The single fibre strength of the pulp was positively correlated with the degree of polymerization, indicating that the autoclaving environment caused a significant decrease in the single fibre strength of wood pulp, which would subsequently lead to a decline in the overall performance of the pulp (Biermann. 1996)

The aforementioned results suggest that wood pulp in fibre cement products exhibits a tendency towards degradation in an alkaline environment, while the damage to pulp properties in an autoclaving environment was more significant. This was primarily evidenced by the slow dissolution of effective components such as cellulose, lignin, and hemicellulose within an alkaline environment, coupled with a significant decrease in fibre polymerization degree under autoclaving conditions, ultimately resulting in the degradation of fibre properties. Therefore, reducing the alkalinity within the interfacial area between wood pulp fibre and cement matrix, along with lowering the autoclaving temperature, can effectively protect the wood pulp and enhance their durability in fibre cement products (Song Junlong, per.comm.).

DISCUSSION

If the first transformation of the fibre cement industry was from asbestos products to non-asbestos ones, it is now confronting the challenge of undergoing a second transformation. The low-carbon economy and sustainable development goals have emerged as irreversible global development strategies, imposing new requirements on traditional fibre cement formulations and production processes. The adoption of low-carbon cement, highactivity reinforcement materials, and recycled fibres could potentially represent promising new technological avenues for the fibre cement industry.

Based on the research presented in this paper, Microsilica plays a crucial role in enhancing the strength of fibre cement products and improving the interface performance between PVA fibres and substrates. The high pozzolanic properties of Microsilica enable it to react with calcium hydroxide at room temperature, producing more C-S-H cementitious materials that contribute to strength, thus making it possible for autoclaved fibre cement products to reduce the autoclaving temperature. Additionally, the reaction to consume the calcium wood pulp fibre degradation too.

CONCLUSION

Wood pulp fibres and PVA fibres are the main reinforcing fibres in non-asbestos fiber-reinforced cement products. PVA fibres have a crucial impact on the toughness of the products.

Wood pulp fibres are at risk of degradation in an alkaline environment. Autoclaving conditions will increase the risk of degradation and significantly reduce the performance of wood pulp fibres.

Microsilica has a significant reinforcing effect on fibre cement products. The reinforcing effect is primarily due to its pozzolanic characteristics, which consume calcium hydroxide to produce more cementitious materials, thereby enhancing the matrix's strength. Additionally, reducing calcium hydroxide helps to decrease the alkalinity near the fibres, which is beneficial for delaying the degradation of wood pulp.

Microsilica significantly improves the interfacial properties between the fibres and the matrix, thereby effectively increasing the contribution rate of the fibres.

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APPENDIX

Specification of tested PVA fibres, wood pulp and Microsilica.

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Specification of PULP 2

Kamloops, British Columbia Canada KAMLOOPS TRUFLEX is a premium, low brightness softwood pulp that is manufactured using selected residual chips from sawmills in the interior of British Columbia. The primary wood species for this grade are white/Engelmann spruce and lodgepole pine. The fiber length and low coarseness result in excellent sheet strength and uniformity. Typical Response to PFI Laboratory Refining Test Method **Initial** PFI Refined 715 500 400 Canadian Standard Freeness (ISO 5267-2 680 650 300 15 $15 22$ 31 42 $14 -$ Schopper-Riegler ISO 5267-1 ISO 5264-2 890 2380 6210 8640 11600 PFI Revolutions $\mathbb O$ number Strength $7,0$ 8.9 9.7 10.5 Tensile (Breaking Length) 214 ISO 5270/1924 2.8 5.0 km 9.5 Tear Index $(1 - ply)$ mN-m^{2/}g ISO 5270/1974 10.4 14.6 14.1 11.7 10.3 ISO 5270/2758 7.7 1.3 4.6 6.3 7.2 kPa·m2/g 3.0 Burst Index 而十石 Sheet Structure and Aesthetics Bulk **松尾** 1.30 1.47 1.38 1.33 度 cm^{2/g} ISO 534 1.77 1.57 0.68 0.73 0.75 0.77 $g/cm³$ 0.57 0.64 5.0 Roughness (Parker, H10) Lim ISO 8791-4 7.2 6.5 5.8 5.2 4.9 28 45 90 $\overline{2}$ 4 ISO 5270/5636-5 Air Resistance (Gurley) 层4 s/100 mL **Test Method** Typical Pulp Characteristics
Brightness (ISO)*, % 日序
Moisture content*, % 名水量 ISO 3688/2470 $27 - 28$ **ISO 638** 13 Chemical Properties
DCM Extractives, % / 季 男/ / 方 Tappi T204 0.06 **ISO 1762** 0.80 Ash Content (525 °C), % 人仔 Fibre Analysis (OpTest FQA) Handsheet at Initial Conditions ISO 16065-1 2.53 Average Length (LWAFL), mm $-100 \, \text{µm}$. ISO 23713 14.2 Coarseness, mg/100 m (4) Population, million fibers/g ISO 16065-1 5.5 **Bale Characteristics** 86x78x38 L x W x H, cm 230 Bale weight, kg Refined to Approximately 7 km Tensile These are NOT pulp mill specifications. -100 nm $-$ * Mill Test, measured at time of production. 8/18/06 和鱼 Kamloops, BC Canada

Grade Profile KAMLOOPS TRUFLEX

ISO 9001-2000 Registered Certificate #0031323 000683 ISO 14001-2004 Registered Certificate #0031324 000683 PEFC Annex 4 - Chain of Custody Forest Based Products

Domtar

