Ageing behaviour of cellulose fibre cement composites in natural weathering and accelerated tests

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Synopsis The ageing behaviour of two types (naturally cured and autoclaved) of cellulose fibre reinforced cement products have been evaluated independently and compared. Two accelerated ageing test methods have been studied and direct comparisons drawn with ageing in natural weathering. In essence it has been found that the accelerated ageing test method in a CO_2 rich environment appears to simulate the ageing mechanism found in natural weathering, and can be used successfully for durability studies concerned with cellulose fibre products. The ageing behaviour of the products studied may be summarised as an increase in flexural strength and E-modulus of the composite with age. It is suggested that this change in mechanical property development may be a result of, amongst others, carbonation of the matrix, increase in the interfacial bond and change in the reinforcing potential of the cellulose fibres with age.

Keywords Fibre cement composites, cellulose fibres, autoclaving, weathering, accelerated tests, flexural strength, elastic modulus, density, carbonation, environmental tests, composite materials, mechanical properties.

INTRODUCTION

It is well known [1, 2] that cellulose fibres are particularly sensitive to alkali attack. The use of cellulose fibres in cement based composite has, however, been investigated in great depth [3–5] despite their apparent sensitivity to the high alkalinity of the cement matrix. Investigations concerned with the use of cellulose fibres in cement composites would suggest that the mechanism of the ageing process could be directly related to the type of matrix, porosity, fibre type and ageing method used.

In the study of the ageing mechanism of cement composites, three main topics are identified and addressed:

(a) Characterisation of the effect of natural ageing and various accelerated ageing treatments on the changes in mechanical properties.
(b) Characterisation of the effect of the various ageing conditions on changes in the properties of the fibres, and to determine to what extent such changes affect the performance of the composite.
(c) Characterisation of the various ageing conditions on the fibre-matrix interface to resolve the influence of interfacial effects on the long term performance.

This paper should be regarded as an introductory paper which is linked to two subsequent papers [6, 7] dealing with the mechanisms of ageing in two types of cellulose fibre reinforced composite materials cured in a normal environment and in an autoclaving process. The products were subjected to two types of accelerated ageing tests and a direct comparison was made with natural weathering. The development of the mechanical properties, chemical analysis and analysis of the fibre properties are described in this paper. The papers to follow in this sequence are devoted to the microstructural aspects.

EXPERIMENTAL DETAILS

Two products were manufactured on a standard Hatschek machine used in the asbestos cement industry. These were as follows:

(i) Product A – 8% cellulose fibres in a portland cement based matrix and cured at ambient temperature and relative humidity
(ii) Product B – 8% cellulose fibres in a portland cement and silica based matrix and autoclaved

The products were exposed to natural weathering for 5 and 4 years respectively. Similar products were also subjected to two types of accelerated ageing tests. The two test methods were compared with the aim of establishing which test procedure simulated closest the natural weathering conditions. One accelerated test was carried out in a normal environment and the other in a

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Received 15 November 1988 Accepted 16 January 1989

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0262-5075/89/11203093/$02.00

CO_2 rich environment. The dimensions of the flat sheet products exposed were 600 × 400 × 6 mm for natural weathering and 200 × 100 × 6 mm for accelerated ageing tests.

The accelerated test methods were based on wet. dry cycling occurring in natural weathering. The test cycles were as follows:

Test in an ambient environment and at elevated temperatures

Test Cycle:	9 h	submerged under water at 20 °C
(24 h)	3 h	in air at 20 °C
	9 h	infra red radiation 80 °C in air
	3 h	cooling down to 20 °C in air

Test in a CO_2 rich environment and at elevated temperatures

Test Cycle:	8 h	submerged under water at 20 °C
(24 h)	1 h	in oven at 80 °C
	5 h	in oven at 20 °C in a saturated CO_2 environment
	9 h	in oven at 80 °C
	1 h	cooling down from 80 °C to 20 °C

The test cycles chosen were optimised by trial and error experiments based on the degree of carbonation and water penetration of the products. The development of the mechanical properties of the products was measured at various stages of ageing using a standard 3 point bend test procedure (ISO 39611 – 1980 (E)). The density of the products was determined according to standard procedures (achimedean method). The degree of carbonation was measured using a chemical analysis procedure described in reference 8 and this has been expressed in terms of the CO_2 content of the products.

The degree of polymerisation (DP) of the cellulose fibres was measured using a particular test procedure described elsewhere [9]. In essence, the composite is crushed to a powder and the cellulose fibres are dissolved out of the powder using a solution of cupriethylenediamene (CUEN). This method is rather complex since great care has to be taken not to damage the cellulose fibres.

An additional experiment was conducted on the cellulose fibres (pinus radiata) whereby the fibres were subjected to alkali attack at elevated temperatures. This resulted in a break down of the DP of the fibres. The fibres subjected to this treatment were tested for their tensile strength using a standard procedure used by the paper manufacturing industry i.e. the zero span test (Tappi method T231 cm – 85). In essence this test method consisted of diluting the fibres in a large quantity of water and subsequently stirring until a uniform mix was achieved. The water was then sucked off through a filter and the fibre mat formed on the filter was dried. A small portion of the fibre mat was cut out and placed between the jaws of a tensile testing rig. The mat of fibres was clamped between the jaws of the equipment at 'zero span' and pulled apart.

RESULTS AND DISCUSSION

Development of composite properties in natural weathering

The development of mechanical, physical and chemical properties with age was evaluated. Figures 1 and 2 represent typical data of flexural strength and E-modulus of the two classes of materials. There appears to be a general increase in strength and stiffness of the products after exposure to natural weathering. For naturally cured products this increase in strength may be associated with an increase in density from 1750 to 1870 kg/m^3 over a period of 5 years. The extent of carbonation of the composite given in Table 1 is represented by the CO_2 content. This indicates a contribution to the strength increase in the product. For autoclaved products the increase in density over a period of 4 years was 1610 to 1790 kg/m^3. The products were also well carbonated and again would indicate that this contributed to a strength increase in the composite.

The DP of the cellulose fibres in the naturally aged products was found to decrease with age by about 20% for normal cured products over a period of 5 years natural weathering and 35% for autoclaved products. This is in contrast to the strength trends, which show an increase with age. Thus, the weakening effects which might have been expected on the reduction in DP are apparently more than compensated by other processes such as carbonation and densification. It will be pointed out later [6] that the ageing mechanism in cellulose fibre composites is complex and there appears to be a change in the reinforcing potential of the cellulose fibres with age. This would then imply that the measured drop in DP of the cellulose fibres with age is of little consequence to the reinforcing properties of the fibres.

Comparison of natural weathering with accelerated ageing tests

The trends in the material property development during natural weathering have been compared with the trends found in the two types of ageing tests described earlier. The results given in Table 1 suggest that the development of mechanical properties of product A when exposed to an accelerated test with CO_2 rich environment simulates more closely the behaviour in natural weathering. Accelerated ageing in a CO_2 rich environment and natural weathering both led to an increase in strength and E-modulus. Also, the increase in the degree of carbonation for the specimens in a CO_2 environment, compares favourably with the naturally weathered products. Although there appears to be an increase in degree of carbonation in the accelerated aged (normal environment) specimens, this does not correspond with an increase in flexural or tensile strength. The mechanism for this will be discussed later [6]. The increase in density of the product with age may be associated with carbonation of the matrix.

With respect to the cellulose fibre properties, there is a significant breakdown in the molecular chains of the fibres with age, which may be directly correlated with the decrease in DP. This is evident for all three ageing tests.

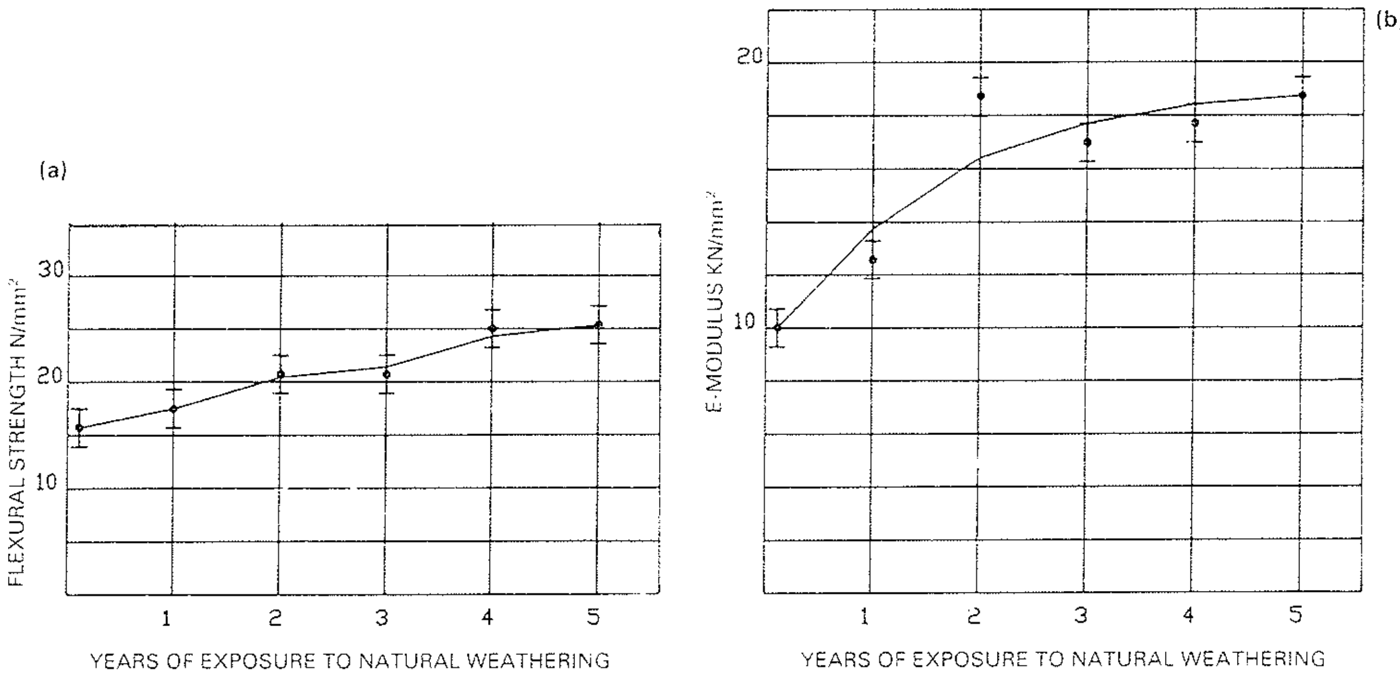

Figure 1 Development of (a) flexural strength and (b) E-modulus of naturally cured products during exposure to natural weathering

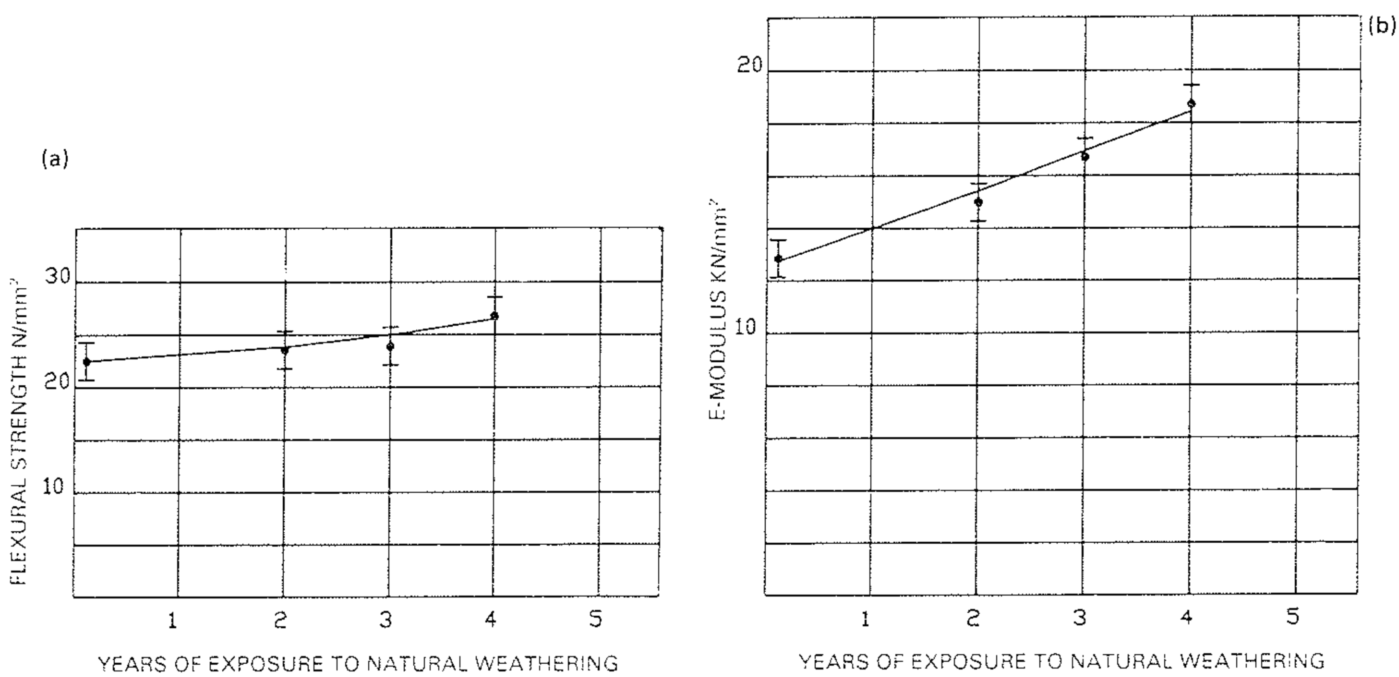

Figure 2 Development of (a) flexural strength and (b) E-modulus of autoclaved products during exposure to natural weathering

It is, however, not exactly clear how significantly the decrease in DP affects the mechanical properties of the composite with age. The drop in DP should logically result in a drop in tensile strength of the cellulose fibres. This aspect may be illustrated by the results given in Table 3 where it is shown that the tensile strength of the fibres (as measured by the zero span test method) decreases in proportion with the drop in DP of the fibres. However, as will be pointed out later, there appears to be other factors, such as the carbonation of the matrix, which may influence the development of the composite with age.

Table 1 Properties of the naturally cured cellulose fibre cement products (Product A) exposed to varied ageing tests

Type of ageing	Flexural strength N/mm²	E-modulus kN/mm²	Tensile strength N/mm²	Strain at failure %	CO_2 content %	DP (fibre)	Density kg/m³
Non-aged	16.4 ±0.9	10.9 ±1.3	7.0 ±0.2	2.80 ±0.70	1.65 ±0.12	581 ±60	1770 ±20
Accelerated aged 3 months ambient environment	12.3 ±1.1	14.8 ±1.2	2.6 ±0.9	0.04 ±0.01	4.85 ±0.16	430 ±50	1780 ±10
Accelerated aged 3 months CO_2 rich environment	23.9 ±2.2	18.9 ±1.4	7.2 ±1.3	0.05 ±0.01	6.71 ±0.05	435 ±80	1800 ±10
Natural weathering	25.1 ±1.6	18.0 ±0.9	7.4 ±0.9	0.05 ±0.01	9.72 ±0.10	491 ±50	1830 ±40

Table 2 Properties of the autoclaved cellulose fibre cement products (Product B) exposed to varied ageing tests

Type of ageing	Flexural strength N/mm²	E-modulus kN/mm²	Tensile strength N/mm²	Strain at failure %	CO_2 content %	DP (fibre)	Density kg/m³
Non-aged	23.0 ±1.3	13.2 ±1.6	10.1 ±1.3	0.07 ±0.02	1.30 ±0.06	730 ±50	1610 ±20
Accelerated aged 3 months ambient environment	20.2 ±1.8	13.5 ±1.8	7.8 ±1.2	0.05 ±0.01	1.62 ±0.21	530 ±20	1700 ±30
Accelerated aged 3 months CO_2 rich environment	25.6 ±1.3	13.1 ±1.1	11.2 ±1.3	0.05 ±0.02	10.7 ±0.22	504 ±60	1790 ±10
Natural weathering	27.2 ±1.4	18.6 ±0.3	12.2 ±1.1	0.04 ±0.01	11.8 ±0.08	475 ±40	1790 ±20

The material property development of the autoclaved product (product B) subjected to the varied ageing tests are given in Table 2. The trends found with these products are similar to those observed for the normal cured composites, the only difference being that the absolute values are not the same and the significance of the trends observed is less pronounced for the autoclaved products. An explanation for this will be elaborated later [7].

CONCLUSIONS

1. The exposure of cellulose fibre cement composites (naturally cured and autoclaved) to natural weathering led to an overall increase in strength and E-modulus with age. This increase was dependent on the type of composite tested.

2. From the results presented in this paper, accelerated ageing in a CO_2 rich environment for 3 months simulates

Table 3 Tensile strength as measured by the zero span test method and corresponding degree of polymerisation (DP) of cellulose fibres (Pinus Radiata)

DP (CUEN Method)	Zero Span (km)*
1630	16.9
810	11.5
810	11.5
870	11.5
270	2.8

* Measure of tensile strength: values are qualitative

closely the chemical and mechanical property development of the 5-year-old naturally weathered product discussed in this paper.

3. The accelerated ageing test method in a CO_2 rich environment is considered to be a more accurate assessment of durability of cellulose fibre cement products than the accelerated ageing test method in an ambient environment without CO_2.

4. The ageing trends observed here (i.e. increase in strength and E-modulus) can not be correlated with changes in the DP of the cellulose fibres. This suggests that there could be other factors contributing to ageing mechanisms which influence the development of the composite.

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