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RECENT DEVELOPMENTS IN DURABILITY OF NATURAL FIBRE CEMENT/CEMENTITIOUS COMPOSITES- A REVIEW

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ABSTRACT

Natural fibre cement/cementitious (NFC) composites has been gaining popularity worldwide due to its potential applications in low cost construction activities. Though there is wide range of opening for NFC Composite, their long term performance i.e. durability under various *exposure* conditions is still a question with unsound answer. Many of the researchers have been working since several decades to overcome this particular issue by providing a right technology for making NFC composites, a really potential product under various applications. This critical review shows a torch on various mechanism of degradability of natural fibres (NF) and NFC composites under different exposure conditions, evaluation of durability of natural fibres and NFC composites, techniques adopted for enhancing durability of NF and NFC composites. The durability improvement is found to be superior with the composites containing cementitious material than in the plain fibre cement composites. Moreover, incorporation/use of modified fibres in the modified matrix shows better performance under durability testing. However, many more studies need to be progressed to confirm the present developments on durability of NFC composites.

Keywords: cement, cementitious, composites, durability, natural fibres.

1. INTRODUCTION

Natural fibres can be proudly termed as a classical material because of its usage for human needs are abundant since from ancient times. Particularly incorporation of natural fibres in cement based materials gained interest from 1960's and due to its potentiality in terms of their inherent properties provoked several researchers to fix the milestones by making use of such fibres in various targeted applications in construction industry.

In general, fibres in concrete are necessary to overcome the inherent deficiencies of concrete such as brittleness, poor tensile behaviour, porous nature etc. Fibres are generally classified as low modulus and high modulus fibres. Among which, natural fibres comes under low modulus category, which are more efficient in energy absorption. Additionally, natural fibres are preferred for its other factors such as low cost, availability, less energy consumption for their production, renewability and non-hazardous. Moreover natural fibre incorporated in cementitious composites shows better performance in terms of flexural strength, toughness, fatigue, ductility and post cracking strength than the plain cement mortar or concrete.

Though the natural fibre composites find its wide applications in construction activities, their performance in terms of long term durability need to be taken at most care to serve its purpose under various exposure conditions. Gram is the first person who made a systematic investigation on the durability performance of natural fibres in concrete. His research findings strongly put forth the point that the natural fibres are not safe under alkaline media such as cement mortar or concrete and thus losing its reinforcing property and ultimately durability of composite gets affected.

Several authors studied the behaviour of various natural fibres such as coir, jute, sisal, bamboo, hemp, wood pulp etc. by incorporating in cement/cementitious based composites. All these fibres contribute to the degradation of the composites particularly made with plain cement mortar or concrete. Degradation of natural fibres in cement based composites occurs because of alkaline pore water dissolves the components of fibre and so its reinforcing capacity is reduced or even completely lost [1]. Also, external environment plays vital role in embrittlement of the composite. It is found that the decomposition of fibres takes place rapidly at high temperature [1]. Moreover other factors contributing to the decomposition of fibres embedded in composite are environment, humidity and also the medium of immersion [2].

It is clear from the fact of previous research, the contribution of both alkaline media and external environment, where the composite is exposed plays major part in degradation mechanism. This condition makes less interest in NFC composites but despite this drawback, the recent investigation encourages the construction industry to make use of NFC products for various applications. Natural fibre concrete products like sheets and boards are light in weight and are used in roofing, ceiling and walls for low cost housing projects [3]. Two major applications based on fibre content are termed as low volume fibre products (eg: Thin roof sheets) and high volume fibre products (eg: Roofing tiles) has been investigated and suggested as potential products [4]. Also in some African countries, sisal fibre reinforced concrete is extensively used as tiles, corrugated roofing sheets, pipes, gas tanks, water tanks and silos [5]. In Australia, wood and sisal fibre reinforced concrete is being used for panels and building boards [5]. It is well known that the natural fibres are low modulus and thus it can absorb more energy which in turn produces composite with higher toughness i.e. it can take more impact loads. Hence NFC composites are utilized where the impact loads are expected to be very high. Many

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more applications of wood-fibre cement in industrial, commercial and residential areas are found in sound proofing, laboratory tops, modular flooring, duct lining, flat and corrugated sheet- roofing elements, exterior and interior wall panels, substrates for tiles, window silts, stair treads and risers and cladding panels [6].

The wide variety of above applications of NFC composites needs to be achieved by producing the composites with high potentiality under long-term exposure conditions. However, the successful performance of composites lies in enhancing the durability of fibres inside the composites [1, 5, 7] when exposed to various external environments. From the extensive investigations, it is evident that durability of NFC composites can be enhanced by adopting various treatment methods and technologies. Gram suggested that surface modification of fibres and matrix modification proves to achieve durable composite. Fibre surface modification has been done with impregnating agents such stearic acid, formine, barium nitrate, natural resins, silica slurry, silane etc. and matrix modification by partially replacing OPC with pozzolanic materials such as silica fume, slag, fly ash, metakaolin etc., using alumina cement, addition of gypsum, sealing matrix pores with natural resins, tannin and wax. Replacement of 45% of cement with silica fume eliminated the loss of toughness of composite and pre-treatment of fibres with water repellent agent retarded the embrittlement of composites [1]. Use of pulped fibres in composites also shows improved performance under durability tests.

2. THREATENING ISSUES OF NATURAL FIBRES IN VARIOUS CONDITIONING MEDIUM AND COMPOSITE PRODUCTION

Natural fibres are now gaining attention in reinforcing cement/ cementitious composites mainly for its potential reinforcing capacity and low cost. Natural fibres are also referred to as vegetable fibres or plant fibres. Fibres are classified based on the parts of plant from where it is actually extracted. They are bast or stem fibres (eg: jute, mesta, banana etc.), leaf fibres (sisal, pineapple etc.) and fruit fibres (eg: cotton, coir, oil palm etc) [8]. Moreover natural fibres are of varying nature have several threatening effects and issues by its inherent characteristics, which has been discussed below.

The physical properties of natural fibres vary widely for different fibres. From one of the study, it is concluded that the coir fibres are thick in middle with tapering ends and their cross sections are oval or circular in shape. Jute fibres on the other hand have rough polygonal cross section and these vegetable fibres are examined under large magnifications, say 1000, it is revealed that they have several cavities and some of them extend to the entire length [9].

Natural fibres extracted from plants are considered as composites consisting mainly of cellulose fibrils embedded in lignin matrix (resin) [8, 10, 11]. The main chemical constituents of natural fibre are cellulose, hemi-cellulose, lignin and pectin whose composition varies from one fibre to other [1, 10, 12]. A single fibre of all natural fibres is made up of several fibre cells. The fibre cells are linked together by means of middle lamellae, which are made up of hemicelluloses, lignin and pectin and are known as cementing material. The fibre cell in turn consists of number of walls made up of fibrillae. These wall layers are namely outer primary wall, outer secondary wall (located inside the primary wall), inner secondary wall and innermost tertiary wall. The fibrillae of different walls composed of cellulose molecular chains [1]. The structure of fibre cell is shown in Figure-1. The important factor contributing strength and reinforcing capacity to the natural fibres are degree of polymerization (DP), spiral angle or microfibrillar angle, chemical compositions and cell structure. The DP of the cellulose of sisal fibre is about 25000 which is less than the hemicellulose, whose DP lies between 50 and 100 [1]. In general, DP of cellulose is 10 to 100 times higher than that of cellulose [13]. Moreover, natural fibres with higher DP of cellulose, longer fibre cell length and lower microfibrillar angle possesses higher mechanical strength and vice-versa [14, 15]. According to Gram, microfibrillar angle of various fibre cell walls varies from 18^o to 40^o for a single sisal fibre. On the other hand, microfibrillar angle of coir fibre is 49^o and for jute is 8^o [10].

Addition of natural fibre in cement composites will poses several negative effects on the composites. These effects may affect both fresh as well as hardened properties of the matrix. The various degrading characteristics of natural fibres such as coir, sisal, malwa, surgarcane, jute, hemp etc., have been evaluated in previous investigations. The major negative issues summarized regarding natural fibres are balling effect, embrittlement of fibres, water absorption, poor bond due to swelling or shrinkage and poor slump and workability [1, 7, 16].

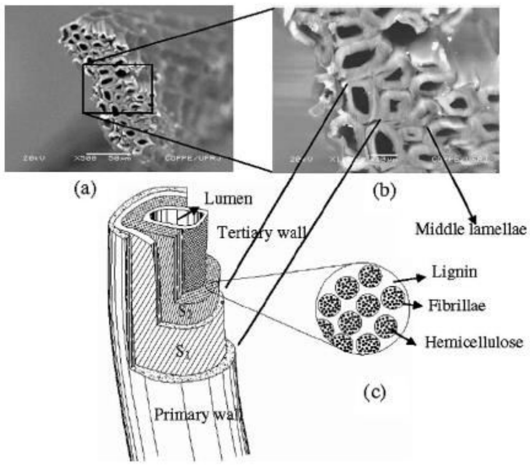

Figure-1. Fibre cell microstructure; (a) cross section; (b) Magnified cross section and (c) individual fibre cell [17].

During composite production, balling of fibres contributes much effect on composite fresh characteristics. The bundling and interlocking of fibres in one particular place in the matrix is known as balling of fibres. There are several factors responsible for balling effect namely aspect ratio of fibres, fibre density, fibre content, size of aggregate used, water-cement ratio and method of mixing [5, 18]. Thus the balling effect in turn affects the workability of the mix and causes insufficient distribution of fibres inside the matrix which diminishes the reinforcing capacity of fibres and thus makes the purpose of fibre reinforcement in the composite to be unresolved. Hence, it is important to have uniform and random distribution of fibres in the matrix during mixing [5]. From an investigation, it is inferred that the flow values reduces with increase of fibre content in the sisal fibre mortar and thus it is clear that fibre addition in the matrix affects the workability of mix. Also, higher the aspect ratio in 1:3 mortar mix shows the reduction in flow value of the mix and thus the reduction is upto 73% for 2% of fibre content which may not desirable from practical applications. Moreover, the irregular surface of natural fibres with several cavities and pores makes the fibres more affinity towards water and causing more absorption of water and this creates various unwanted issues during composite production. Also the large surface area and water absorption of fibres leads to the reduced workability of mix [5, 18]. The water absorption may range from 50% to 200% of dry weight of fibres [9]. It is found that the greater water absorption of fibres upto 95% for malwa and sisal for first 30 minutes when compared with coir fibre [16]. The male date palm fibre presents the water absorption of 132.5% for 24 hours immersion and the fibre has mean porosity of 35% [19]. The water absorption affects the water-cement ratio of composite mixes and additional water needs to be added for fibres or presoaking of fibres has to be done in freshwater for 48 hours and surface dried before mixing will make the mix better workable [5, 19]. The dimensional variation of vegetable fibres, which is due to high water absorption of malwa and sisal, causes insufficient bonding and thereby affecting the composite performance under service [16]. The excessive porosity of fibres contributes inferior macro-structural performance of composites and high water absorption of fibres causes local increase in water cement ratio, which creates the highly porous transition zone and this condition is more pronounced in composite exposed to hot-dry climate [16, 18, 20].

There is another major threatening problem associated with natural fibres is embrittlement of fibres under alkaline environment. It is found that the fibres are deteriorated due to chemical action (i.e. alkaline environment) and biological decay [5]. Exposure of fibres under water for longer period will leads to biological decay of fibres and thus it is found that the sisal fibre stored under water has become red and changes to black with time which indicates the bacterial action on components of fibre [1,5]. But fibre embedded inside cement matrix inhibits biological action and shows good durability against biological decay [5]. The chemical action on fibres is mainly due to chemical decomposition of lignin and the hemicellulose in the middle lamellae or alkali attack and migration of hydration products to lumens and voids of fibres [1]. Due to decomposition of lignin and hemicellulose, which are the cementing materials of the microfibers of fibrillae, the link between individual fibre cells gets weakened and loses its reinforcing capacity [1]. The fibres stored under saturated lime solution with pH 12.6 shows reduction in strength. But fibres stored under prepared pore water of pH about 12.5 and 13 have presented less decomposing effect than lime solution. This proves that lime solution have had more powerful decomposing effect on fibres irrespective of pH of various solutions. The decomposition of natural fibres in alkaline solution is faster than in water and also the decomposition depends upon type of fibre, pH value of exposure medium, medium of immersion and environmental factors such as heat, solar radiation, and humidity [21,22,23]. According to Gram, it is found that the sisal and coir fibre stored under lime solution with temperature of $+50^{o}$ C have lost all its strength after 14 days and this effect is more severe when compared to the fibres under immersion in room temperature. In another investigation, it is concluded that the tensile strength of sisal and coir decreases up to 50% when immersed in saturated solution of calcium hydroxide (CH) of pH 12 for 28 days [23]. Also, it is found that after 210 days of immersion of sisal and coir under calcium hydroxide solution retained only 33.7% and 58.7% of their original strength which have been completely lost after 300 days [7]. The about facts of fibre decay is more evident and significant with the increase of age of immersion [16].

Another issue associated with natural fibres is delay of setting time of the composites. This effect is due to lignin and hemicellulose hydrolysis and effect of water soluble extractives such as sugars present in fibres [24, 25, 26]. The sisal, coir and raw bagasse fibres were studied for their extractives effect on composites. The coir fibre extract has more pronounced effect on setting of cement mix compared to sisal fibre extract [27]. Delay in setting of raw bagasse cement composites are also found to be more than the plain cement mix which is due to the presence of water soluble sugars in bagasse fibre [28].

3. DEGRADABILITY OF NATURAL FIBRE CEMENT/CEMENTITIOUS COMPOSITES

Natural fibre cement/cementitious composite for construction is said to be a successful material only when it possesses better serviceability in addition to strength parameters [18]. The material is said to be serviceable only when it is found to be durable under various exposure conditions where it is actually put into practice. Durability relates to its resistance against degradation/deterioration due to external causes such as effects of environmental and service conditions such as weathering, chemical actions and wear and internal causes such as alkali-fibre reaction, volume changes, permeability and water absorption [29]. Natural fibre composites are more vulnerable against degradability than other synthetic fibre composites. Both inherent properties of fibres as well as the environment, where the natural fibre composites are exposed impart to the degradation. Various mechanisms are responsible for a composite degradation and those mechanisms depend upon various factors, which lead to different forms of failure under service.

3.1 Factors contributing to the degradability of NFC composites

Several degradation factors are responsible for the deterioration of a natural fibre composite. Various factors considered and durability has been ascertained by various investigators are mostly based on fibre characteristics alone. But in real considerations effect of exposure conditions to which the composites are exposed are also equally plays a role against durability of composites [30]. In realistic, the various contributing factors for degradation of natural fibre composites are fibre characteristics (fibre type, fibre content, fibre geometry, fibre surface, fibre alignment and fibre form), matrix properties (matrix type, cement type, aggregate type and grading, additives/admixtures and water-cement ratio), composite casting techniques (mixing method, placing method and curing methods), composite product properties (size, shape, type of application) and exposure conditions (laboratory medium, real environmental/ climatic conditions). Among above factors, the significant and threatening effects are contributed by fibre characteristics and exposure medium. Table-1 summarizes the various above factors in detail.

Table-1. Factors affecting degradability of NFC composites.

Fibre characteristics	
Fibre type (natural fibres)	Coir, sisal, bamboo, malwa, akwara, jute, flax, wood, surgarcane bagasse, date palm
Fibre content	Low fibre content (1-3%) ; high fibre content (> 3%)
Fibre geometry	length, diameter, aspect ratio
Fibre surface	Rough (natural), smooth (due to coating)
Fibre alignment	Long aligned, short discrete
Fibre form	Strands, pulps
Matrix properties	
Matrix type	Cement, mortar, concrete
Cement type	OPC, PPC
Admixtures/additives	Pozzolanos (slag, flyash, silica fume, metakaolin), natural resins, synthetic polymers, super-plasticizers
Other parameters	Water-cement ratio, aggregate type and grading
Composite casting techniques	
Mixing method	Type of mixer, sequence of adding constituents method of adding fibers, duration, speed of mixing
Placing method	Conventional vibration, vacuum dewatering for sprayed-up member, vacuum-press dewatering for slurry-dewatered member, extrusion and guniting
Curing methods	Conventional, special methods
Composite product properties	
Type of application	Roofing sheets (flat/corrugated), roofing tiles, building boards and panels (exterior and interior wall panels), pipes, gas tanks, water tanks, silos, sound proofing, modular flooring
Other parameters	Product sizes and shapes, age of product
Exposure conditions	
Laboratory medium	Tap water, alkaline medium, alternate wetting and drying, alternate elevated temperature and room temperature, alternate elevated and curing in normal water temperature, freezing and thawing, accelerated carbonation, hot water medium
Environmental/climatic conditions	Varying factors related to outdoor environment such as temperature, relative humidity, wind velocity, and conditions of rain, snow and ice

3.2 Mechanism of degradation of NFC composites

The degradation of all NFC composites follows several failure mechanisms for losing their strength and durability. Those mechanisms are mainly associated with the several modes of fibre failure which in turn depends upon ageing process under which the composites are exposed. Different mechanisms are summarized as follows [1, 5, 31, 32]:

a) Alkaline hydrolysis of cellulose molecules results in peeling off mechanism, which occurs at end of molecular chain. The end group is reductive and reacts with hydration products and unhooked from the molecular chain, causing reduction in degree of polymerization.
b) Dissolution of lignin and hemicellulose in the alkaline matrix breaks the link between individual cell fibres. Also, the decomposition of hemicellulose and lignin follows the same pattern of failure mechanism as that of cellulose molecules.
c) Increase in fibre-matrix bond or stress corrosion leads to embrittlement of fibres inside the matrix.

d) Microbiological/microbial attack on fibres under less alkaline matrix.

The different mechanisms responsible for composite degradation mainly depend on external environment and period of exposure to which the composite is exposed. Thus the pore water present inside the mortar/concrete matrix is highly alkaline and it attacks the natural fibre chemically. As the result, lignin and hemicellulose in the middle lamella of fibre decomposes first and thus leads to initiation of reduction in matrix behaviour. On the other hand, external environment plays vital role in maintaining the continuous supply of new alkaline solution to the natural fibre and thus accelerates the decomposition of fibre and makes the composite to behave brittle [1,7,33,34]. Therefore, in macrostructural aspects, the external environment leads to failure of composites by various stages [35] as follows:

a) Loss of ductility with improved strength (i.e. improved interfacial bond due to carbonation, drying and shrinkage).
b) Loss of mechanical properties due to disruption of matrix and debonding of fibres from matrix.
c) Serious damage of composite due to embrittlement of fibre materials and matrix degradation, as a result of continuous exposure to increased moisture movements.

Various failure mechanisms contributes to the several modes of fibre failure based on ageing process of composite, as confirmed by micro structural analysis. Typical modes of failure observed are fibre pullout and fibre fracture [9], former case resembles the ductile mode of failure and later case is brittle failure. The brittle failure exhibits in two different ways and are brittle hollow (due to change in fibre content which is partly filled by cement hydration products) and brittle petrified (due to progress in carbonation, which fills up the voids left in fibres) [31,33]. In brittle mode of failure, the fibre fails close to the fractured surface and fibre seems to be filled up with hydration products or left hollow (i.e. natural form), thus depends on exposure conditions of the composite. The composites exhibiting pullout failure is considered as ductile composite and possesses higher value of modulus of rupture and tensile strength, whereas the composite showing brittle failure contributes to reduction in strength or increase of strength based on nature of fibre, ageing period and type of exposure conditions [31].

3.3 Effects of various degradability factors (exposure conditions and weathering on degradability) on NFC composites

The specimen or element exposed to various conditioning medium and natural weathering are very important in influencing the degradation process and also it is equally important to their effects on composite behaviour. It is very difficult to organize the degradation of NFC composites in particular order because of variability of properties of natural fibres, different degradation mechanism shows different effects and presence of multiple contributing factors for the reduction of long term behaviour of the composites. However, the discussion is presented in such a way to understand the critical failure and degrading patterns of NF composite with respect to various exposed conditions. The different exposed conditions and their effect on natural fibre reinforced composites are discussed critically below.

As mentioned earlier, it is well known that the cement based matrix itself causes decomposing effects on NF composites due to its strong alkaline nature. But severe damage occurs only when the interaction with external environment continues to act on composites, keeping a big question mark over the long term performance if the problem is unnoticed. Gram, who is a pioneer for suggested the systematic mechanism for decomposition of products and he reported that the embrittlement of sisal fibre concrete depends on the magnitude of variations in relative air humidity and temperature to which it is subjected. Thus, this embrittlement closely related to the external environmental conditions. In general, the exposure conditions are considered in two ways Viz. intermittent exposure and continuous exposures for both laboratory as well as natural weathering. The cement based NF composites exposed continuously to dry or wet environment or in any one particular medium is said to be continuous exposure condition and thus the alternate or cyclic conditions prevails for same specimen for particular time intervals are treated as intermittent exposure condition.

a) Continuous water and air curing; Alternate wetting and drying (under normal condition)

Exposing composites to continuous and alternate mediums will have different degrading effects. Thus, the sisal [1] and agave lecheguilla [36] fibre reinforced composites exposed to constant dry environment shows damage with time, but under constant wet environment doesn't show any sign of damage. This effect is due to the fact that under continuous wetting, the transport of OH^- ions proceeds slowly [1]. Also, the flexural and compressive strength of natural fibre matrix is greater under continuous water curing even for later aging but under alternate wet/dry conditions shows poor performance for earlier ages and also lose of reinforcing capacity is very severe in wet/dry conditions than water curing [22,30]. It is observed that specimen stored under water for 322 days exhibited the strain hardening behaviour under load-deflection curves, whereas specimens subjected to 25 to 46 cycles of alternate wet/dry cycles showed strain softening behaviour [7]. Moreover, increase of strength is observed for continuous exposure to both water and air curing but air cured definitely shows poor performance than water cured one, due to the lack of hydration products, which in turn due to rapid evaporation of water in concrete at early age itself [19]. This fact is confirmed by the XRD analysis, showing higher level of C_3S and C_2S and thus proves the reduced level of hydration products [19].

b) Alternate wetting and drying (under accelerated condition)

This type of exposure conditions has more pronounced effect because of exposure to high temperature and freezing/thawing than wet/dry cycles under normal conditions. The decrease of composite strength and severe embrittlement of fibres are observed under accelerated wet/dry cycles. In one of the study, it is noted that sisal fibre concrete showed very serious damage at higher temperatures and is due to increased chemical action on fibre is more when exposed to higher temperatures [1]. In another study, it is reported that the composite presented increased flexural strength failure and initial stiffness but with reduced toughness with brittle [6]. On the other hand, composite exposed to freeze-thaw conditions does not have much effect on the flexural strength and stiffness but showed increase of porosity with increase in fibre volume [6, 37]. In case of water absorption and bond strength, the increase in water absorption and decrease of internal bond are exhibited by the bagasse cement samples after 10 to 25 cycles of wet/dry exposures [34]. SEM images reveal that accelerated alternate wet/dry conditioned specimens possesses dense matrix around the fibres and embrittlement of fibres. Also, it is explained that the fibre seems to be stronger, rigid and brittle and these facts improved the first crack strength (FCS), internal bond strength and lowered the toughness and ductility of composites [7].

As the age and number cycles of exposure increases, the mode of fibre failure has showing the significant effect. In this aspect, the predominant mode of failure noted for composite with less exposure is fibre pullout but specimen after 25 cycles presented fibre fracture due to brittle nature of fibre, which experience repeated swelling and shrinkage and therefore, resulted in mineralization of fibres [6]. The above effects are mainly depends on the failure mechanism such as fibre cement de-bonding during initial drying cycle, re-precipitation of ettringite after 2 wet dry/dry cycles and re-precipitation of calcium hydroxide after exposure to continuous wet/dry cycles [38, 39, 40]. It is threatening to observe that the coir fibre reinforced composite under alternate air and sea water exposure, because of severe expansion and shrinkage which leads to the manual pullout of fibre from fractured specimen. This effect is attributed to the crystallization of salt around the fibre affect the fibre flexibility and strength [41].

c) Hot water immersion

The hot water immersion increased the porosity of cellulose fibre composite after 100 days of exposure with the increase of fibre volume [37]. Also, it is observed that the flexural strength has increased or possesses very small effect on composites, but its behaviour is very different in case of stiffness of composite, showing both increased and decreased effect based on the nature of fibres used [6,37]. As compared with other exposure conditions, hot water immersion caused very least effect on composites behaviour [6].

d) Alkaline medium

Various alkaline mediums adopted are calcium hydroxide, sodium hydroxide [30, 42], sodium chloride, sodium sulphate [36] and sea water [41] in terms of continuous immersion and alternate wet/dry conditions. Reduction in flexural and compressive strength observed in mortar is less and at very slow rate in continuous immersion than in alternate conditions in all immersion mediums [30]. In comparison with all exposure medium, composites exposed to chlorides exhibited nearly 30% reduction from their original strength due to crystallization of salt and fractures due to crystal growth expansion inside the composite [36,41]. Also, the impact strength and flexural strength of sisal fibre composite decreased after exposure to alkaline medium [42].

e) Natural weathering and accelerated carbonation

NFC composites subjected to natural weathering and accelerated carbonation shows almost similar degrading effects. Thus the composite under 25 years of natural weathering shows damages by brittle mode of failure with dense matrix and reduced toughness and strength but unaged specimen exhibited pullout mode of failure with higher toughness and strength [31]. It is noted that fibre taken out from 10 months old sisal fibre roof unit presented two different fibre failures. Thus the fibre from surface is in intact with the matrix and presented the pullout length of 50mm but fibres from centre of section are brittle with pullout length of only 2mm. This effect could be due to the effect of carbonation on the surface of roof unit with low pH than non-carbonated inner section [1]. Also, XRD pattern with low CH and more CC contents is noted for specimens under natural weathering and accelerated carbonation as compared to unaged specimen [31].

On contrary to above fact, the composite subjected to natural and accelerated CO_2 environment leads fibre breaking in fractured plane, representing fibre lumen with carbonated hydration products which leads to reduced fibre flexibility [31, 33, 43]. In macro-structural aspects, the composite shows increased flexural strength and initial stiffness but with decreased toughness even up to 54%. Also chemical analysis proves this effect and the degree of carbonation causes improved strength by densification process and loss of DP of cellulose and lowered lignin content are causing the breakdown of reinforcing capacity of fibres [20, 44]. When E-grandis pulp cement composite aged under both accelerated carbonation and natural weathering are compared, it is noted that the tropical natural weathered composite exhibited more severe effects than in accelerated carbonation due to the cyclic action of temperature and moisture [44].

4. EVALUATION ON DURABILITY OF NATURAL FIBRES AND NFC COMPOSITES

Behaviour of composites can be assessed in confidence by means of studying its strength and durability. The durability assessment is not a simplified way of determining the long term performance of NFC

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products. It needs several interrelating factors to be studied in deep sense for understanding the actual long term behaviour. In case of natural fibre reinforced composites, not only the external environment but also the internal environment in the cement matrix, which is alkaline in nature plays important role in knowing actual facts of deterioration mechanism. The most important aspects in understanding the evaluation of durability of natural fibre composites are exposure/conditioning medium to which the NF and NFC composites are exposed and the respective methods or criteria to test the fibres and composite respectively. The specimens are usually exposed under some environment varies in two ways namely real time conditions and accelerated laboratory conditions.

4.1 Natural fibres

Natural fibres, before incorporating into cement composites are subjected to various exposure medium and tested after conditioning to understand the independent behaviour of fibres with respect to exposure and conditioning medium.

4.1.1 Evaluation of durability of natural fibres

Evaluation of fibre durability is measured after exposing natural fibres in a particular medium for specific durations. The measurement involves in testing of fibres for its degradation and evaluated in terms of change in its typical properties such as dimensional stability, strength and chemical composition of fibres. The dimensional stability of fibres are provided in respect of change in length, diameter and weight of fibres, strength evaluation is based on the fibre's tensile strength and chemical composition with respect to change in content of lignin, hemicellulose and cellulose. These can be ascertained by comparing the properties of fibre in its natural condition (i.e. before being exposed to any medium). Any changes or variation occurred in the fibres subjected to various mediums are clearly determined by mechanical and chemical evaluation.

Tests results of various natural fibres exposed to different medium varies accordingly with respect of distinct parameters of same immersion or exposure mediums. It is reported that pH, temperature, relative humidity and type of immersion are critical parameters, which affects the properties of fibre [1, 30]. In general, the fibres stored in alkaline solutions and chemical medium shows the effect of alkali attacks on fibres, whereas for fibres under water exhibited microbial attacks [1, 7, 30]. Moreover loss in strength of fibres happened irrespective of medium of immersion (i.e. all mediums contributed to reduction in strength). The alkaline activity is responsible for fibre degradation, which implied that as increase in pH of alkaline medium triggers the degradation of fibres. But it is reported that the lime solution (pH=12.6) has more pronouncing decomposition effects than the prepared concrete pore water (pH=13.7). It is clearly suggesting that the importance of type of medium of immersion rather than pH in affecting the fibres [1]. In comparison with other alkaline mediums, calcium hydroxide solution contributes to higher attack, which is due to the crystallization of lime in pores of fibres and so fibres becomes increasingly brittle [1,7,45, 46].

In evaluation process, from above facts it is clear that pH alone is not considered as determining factor for fibre failure. Moreover, considering typical properties of fibres after exposure will provide beneficial ideas in understanding the fibre failure mechanism. In this regard, analysis on dimensional stability, tensile strength and chemical composition are effective in grading the fibre properties. The tensile strength and modulus of elasticity of fibres gets reduced with respect to the type of fibre and type of medium of exposure. The loss in strength for coir fibre stored in lime solution for six months duration shows 65% of its original strength which is less than sisal fibre with only about 20% of its original strength [1]. Considering the fibres such as sisal, banana, coir, hemp and jute exposed to cyclic wetting and drying of alkali solutions exhibited elongation of break, ranges between 10-40% except for hemp and jute, showing brittle mode of failure [46]. The modulus of elasticity of coir and palm fibre showed reduction in its value and is more pronounced for alkaline medium than water medium [5]. Also fibres such as sisal, coir, date palm, jute and hibiscus cannabinus suffered severe degrading effects on fibre dimensions, tensile strength, modulus of elasticity, chemical composition and rate of leaching of extractives, when stored under $Ca(OH)_2$ medium compared to other mediums Viz. NaOH solution, water and pore solution respectively [1, 5, 7, 27, 45, 46].

In terms of chemical composition, there is considerable reduction in chemical composition of natural fibres, contributing losses up to 20-70% of cellulose and 30-70% of lignin with that of its original composition after exposing to alkaline mediums of continuous and cyclic immersions [30]. Another fact to be considered is elevated temperature of conditioning medium which accelerates the alkaline activity and contributes to severe degrading mechanisms as that of mediums at ambient temperature [1]. Moreover, to understand the failure mechanism of natural fibres in critical aspects, single test is not sufficient. Attempting the combination of tests on particular type of fibre with its various interrelated parameters are mandatory, since more varying nature of fibres makes complications in judging the degradation process.

4.2 Natural fibre cement/cementitious composites

Unlike natural fibres, natural fibre cement/cementitious composites are considered to be affected by three phase degradation criteria Viz. environmental conditions (external environment), matrix conditions (internal environment) and the condition of fibre itself. The complications of contributing factors responsible for composite degradation could be undoubtedly numerous and needs precise level of evaluation process.

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4.2.1 Evaluating methods for assessing durability of NFC composites

Assessment of durability of NFC composites will be successful only when systematic test approaches are employed to study the characteristics of composites for finding better applicability of NF products. This could be possible by conducting various strength tests over the composites after exposure and subsequently analyzing the critical test parameters in terms of flexural strength, compressive strength, toughness and impact strength respectively. Each of strength parameters will have independent impacts on composite behaviour and needs deep insights to make natural fibre composites as one of real and promising construction materials.

Thus from above explanation, the durability of natural fibre composites are very closely related to the behaviour of fibres inside the matrix when subjected to particular type of loading. During testing of composite, once cracks appears, the fibre acts as crack arresters and thus absorbs more amount of energy with complete pullout of fibre from matrix [3]. Evaluating this energy absorption and arriving respective interrelating study parameters is one of the important aspect of analyzing the durability aspects of NFC composites. Another easy way of durability evaluation is studying the shape of stress-strain curve, peak flexural stress and post cracking behaviour. Thus, the Fig. 2 shows the various stress-strain curves. The curve A is attributed to the unreinforced matrix. The fibres with high tensile strength and modulus of elasticity exhibit high tough and ductile composites and thus it relates to the curve B. When reinforcing capacity of fibres affected, the curve B falls to curve C or even to unreinforced pattern [47].

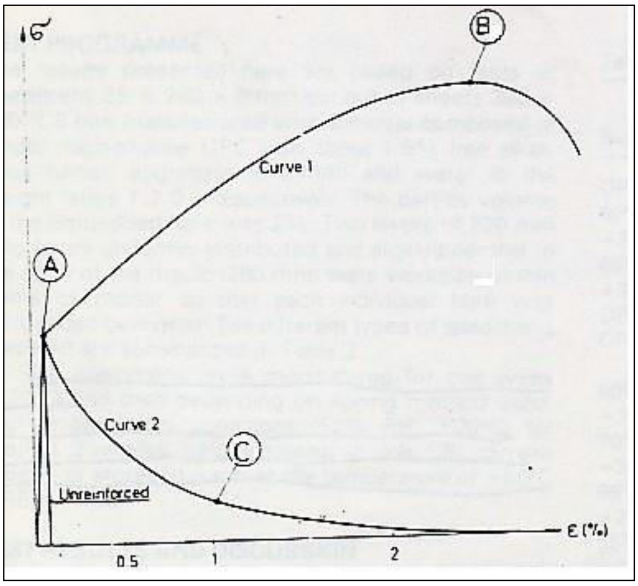

Figure-2. Stress-strain curve of various matrices [47].

In studying the NFC composites, the time and type of aging affects the curve pattern with respect to peak flexural stress and post cracking behaviour [22]. When testing the sisal fibre mortar, it shows that fibre in composite embrittled after 6 months of natural weathering and is proved from flexural strength test [43]. Also the moisture effects on flexural performance of composites are evaluated in terms of strength, toughness and stiffness. It shows that the increased moisture exposure affects the flexural strength and stiffness in negative aspects but increases the toughness of composite. On the other hand, this effect seems to be vice-versa in case of composites exposed to repeated wetting and drying cycles [6]. Evaluating the fracture toughness is closely related to the NFC composite rather than flexural strength parameter, because natural fibre are low modulus and is intended to absorb energy during load application. The fracture toughness in turn related to two different mechanism Viz., fibre fracture and fibre pullout [48].

One of the studies has presented that the sisal and coconut fibre cement mortar shows the reduction in T_{JCI} (Toughness parameter) values by about 53% when aged under water and with load-deflection curves of strain hardening behaviour. But the specimen subjected to outdoors and repeated wet/dry cycles exhibited work softening as its predominant behaviour [7]. In another facet, not only degradation process of composite could be assessed by using post cracking and toughness behaviour but also improvement in composite performance with enhanced durability can be clearly attempted. Therefore, the carbonated samples exposed to water curing and outdoors retained 70 to 93% of its original flexural strength and T_{JCI} values and also demonstrated post cracking ductility behaviour [49, 50]. The effect of various stages of aging in terms of degradability of composites is well addressed using load-deflection curves obtained from bending test. Thus from the kraft pulp composites, it is seen that 43-52% loss of FCS, 50-72% loss of peak strength and 75-98% loss of post cracking behaviour toughness respectively in case of 25 wet/dry cycles. After 2 to 5 cycles, the majority of loss in strength and toughness occurred and composite after 25 wet/dry cycles shows very low performance. Moreover, the load-deflection curves showed work hardening in composites subjected to low number of cycles, whereas composites under more number of wet/dry cycles exhibited work softening behaviour [38]. In another view, it is proposed that the actual durability of NFC composites is not evaluated in simple manner, since the interrelating degrading factors are numerous and are more complicated. Hence, evaluating durability in terms of bend test and compression test alone is insufficient, because the composites also will experience the impact loads in actual practice [42]. Therefore, the assessment could be focused on toughness under impact as well as a flexural load has to be compared. In this view, it is evident that the impact and flexural tests parameters follows same trend, showing reduction in impact and flexural strength after exposure to various alkaline mediums [42].

4.3 Evaluating durability in terms of micro-structural and mineralogical analysis

4.3.1 Micro-structural analysis

The morphological changes in the composites before and after exposure to extreme conditions have been clearly dealt using Scanning electron microscopy (SEM). In detail, the SEM analysis gives more information on degradability of composites in terms of effects on transition zone, different phases and structure of compounds, mode of fibre failure, and nature of porosity of composite and micro cracks etc. Nowadays, the SEM equipped with EDXA (Energy dispersive X-ray analyzer) is widely used for examining the cement, because it provides additional information on the spot elemental composition of the composites. The fibre cement composites after exposure to aggressive medium shows varying effects on composite's micro-structural properties and the causing factors for such effects are revealed with SEM/EDXA. This is confirmed by studying the different natural fibre reinforced composites by various researchers in previous studies. In this respect, the micro-structure of examined malwa-cement paste composite shows the degrading effect of composite when subjected to 180 days under water. The interface of fibre and matrix exhibited debonding effect once the specimen is exposed to dry environment. Also, natural fibres such malwa, sisal and coir exhibits higher water absorption of about 80% and this causes the interfacial transition zone (ITZ) with high porosity and the thickness of ITZ measured to be between 50-100μm [16]. The wheat straw cement boards presented only lesser micro-cracks and straw found to be bonded with matrix before subjected to ageing but fibre separation from matrix is highly noticed for the matrix aged under alternate wet/dry cycles [51]. This debonding effect is also seen in sisal and coir reinforced mortar before subjected to wet/dry cycles due to high matrix porosity and exhibited more cracks, but the same matrix after exposed to 25 cycles of wetting and drying, causing opposite trend to that of unaged sisal and coir fibre mortar. This leads to the macroscopic behaviour of the composite with more strength and lesser ductility [7]. The microstructure of the composite stored under two different environment namely in water and hot-dry conditions exhibited opposite observations. Therefore, the date palm fibre composite in hot-dry environment shows more voids and fibre seems to get affected than the composite stored under water medium, which shows the adhesion of matrix and fibre due to penetration of calcium hydroxide and leads to reduction of voids between matrix and fibre [44, 52]. Apart from the composite degradation, the conditions of fibre under various exposure mediums are also clearly presented with different modes of fibre failure under SEM examinations. It is observed that the microstructure in general presented the three different modes of fibre failure viz. ductile pullout, brittle hollow and brittle petrified. The ductile pullout mode of fibre failure is attributed to the unaged cellulose composites, brittle hollow is predominant in aged composite under normal environment and brittle petrified mode of failure is noticed for composites aged under accelerated CO_2 rich environment and natural weathering of 5 years [31]. The kraft pulp fibre from composites subjected to 25 wet/dry cycles presented fibre fracture as the predominant mode of failure but unaged composites presented pullout fibre failure. This fibre fracture effect is due to deposition of hydration products on the fibre surface, which is confirmed by EDXA analysis [38]. The pullout and fracture modes of fibre failure are attributed to the following three mechanisms namely fibre-cement debonding, subsequent reprecipitation of hydration products in the voids of fibre-cement interface and the fibre embrittlement due to mineralization and the degradation is seen in Figure-3. These mechanisms of fibre degradation are well addressed from the microstructure images obtained from SEM analysis [38, 39]. The date palm fibre exposed to different alkaline solutions of calcium hydroxide and sodium hydroxide has exhibited two different micro-structural behaviours. The fibres immersed in $Ca(OH)_2$ solution, the attack of $Ca(OH)_2$ is uniform throughout the fibre but the fibres stored under NaOH solution shows local penetration of solutions into cells and pores of fibre [45]. Moreover, the natural weathered composites presented severely affected microstructure than that of composites exposed to laboratory conditions, showing the severity of natural weathering due to additional mechanism of carbonation [53]. It is evident that the 5 years natural weathered cellulose fibre presented completely carbonated microstructure. The natural weathering of autoclaved cellulose fibre composite showed a microstructure having tobermorite and calcium silicate hydrate (C-S-H) becomes carbonated and rehealing of cracks by calcium carbonate [31, 33]. Another study showed that the coir fibre reinforced concrete exposed to 18 months in tropical climate presented micro cracks at the matrix and gap between fibre-matrix interfaces [41]. Fique fibre cementitious roofing tiles exposed to 14 years in natural weathering shows severely damaged microstructure than that of laboratory conditioned tiles. The natural weathered tiles showed reprecipitation of calcium both in fibre surface and fibre lumen [53]. Eucalyptus pulp reinforced cementitious composites presented more number of pullout of fibre from matrix for unaged non-carbonated samples and fibre fracture failure for one year natural weathered composite under SEM investigations [54].

The improvement or changes in microstructure are also clearly noticed when the natural fibre reinforced composites manufactured by adopting durability enhancement techniques [see section 5]. From the previous studies, the matrix modification, fibre modification and combined fibre and matrix modification are possibly adopted to enhance the durability of natural fibre composites. The fibre modifications such as pulping, chemical surface treatments, bleaching, beating etc., showed improved microstructure than that of unmodified fibres. Therefore, the mechanically pulped sisal fibre cementitious composite shows good bonding between fibre and composite and also presented the interfacial area without any porosity and portlandite concentrations in OPC composites [52]. Also kraft pulping along with

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bleaching removed the lignin, which leads to rougher surface of fibre contributing for increased fibre-matrix bonding as compared to unbleached kraft fibres after subjected to wet/dry cycles [39]. SEM with EDXA showed that the thermo mechanical pulp (TMP) fibre composites with no evidence of fibre inter layer debonding. Due to intact of inter layers of fibres, TMP composites showed no sign of ettringite reprecipitation [39]. In another study, it is seen that the chemically modified cellulose fibre cement composite after 200 wet/dry cycles doesn't show any Calcium hydroxide reprecipitation in fibre lumen and therefore leads to better macroscopic behaviour in terms of higher toughness and deflection. This is also proved by EDXA data and this shows that concentration of carbon atom on fibre cell wall and this plays important role in mitigating the degradation of treated fibres [55]. On the other hand, matrix modification of composite with secondary cementitious material (SCM) such as silica fume, GGBS, flyash, metakaolin etc., improves the microstructure and enhances the composite durability. It is evident that the sisal and coir fibre extracted from the matrix modified with blast furnace slag (BFS) doesn't show any sign of damage and also pulped banana fibre composite after 12 months of exposure to tropical climate showed that the fibre was in good condition without any mineralization and pullout failure was seen to be predominant [44,49]. Moreover, the kraft pulp fibre with matrix modification by silica fume, metakaolin, fly ash and slag showed no sign of leaching of alkalis after 25 wet/dry cycles and this is evident from SEM/EDXA analysis [56]. Sisal fibre reinforced composites with metakaolin also indicates no sign of mineralization but fibre in OPC composites are found to be mineralized due to high concentration of calcium after ageing under hot water for six months. The metakaolin composite exposed to 25 wet/dry cycles also does not shows any degradation effects but the composite without metakaolin presented mineralization of fibre cells due to high calcium hydroxide [40,57].

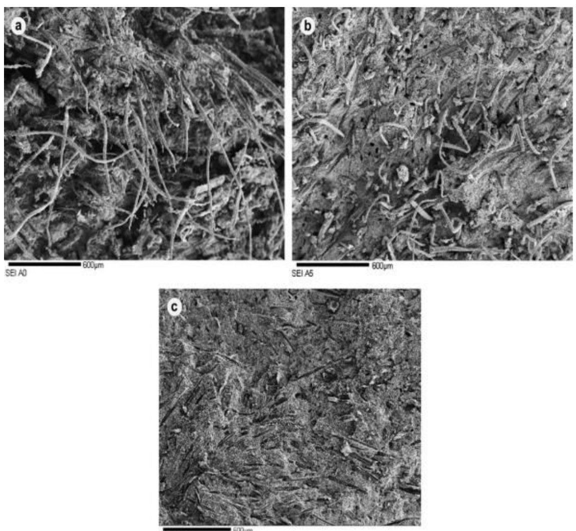

Figure-3. Progression of fiber degradation. (a) Without cycling (b) After 5 cycles (c) After 25 cycles [38].

4.3.2 Mineralogical analysis

The elemental or compound compositions of hydrated cement composites are determined with the help of widely used techniques such as XRD and thermal analysis. Thermal analysis uses two aspects in evaluating the minerals such as Differential Thermal Analysis (DTA) and Thermogravimetric analysis (TGA). DTA provides the thermal peaks to notify the particular element and TGA provide the information with respect to weight loss. On the other hand, XRD quantifies the hydration products such as C-S-H, calcium hydroxide and also several other compounds. It is seen from the study that the autoclaved cellulose fibre reinforced composite before ageing consists of mixture of C-S-H and tobermorite needles and these compounds are found to get carbonated and micro cracks are filled with calcium carbonate after natural weathering for five years. This is also supported by TGA data, which shows high calcium carbonate and low CH content in terms of weight [31, 33]. The degradation effects of date palm fibre concrete cured in water and hot dry environment for three months shows the presence of high C3S and C2S in dry environment as the result of lower hydration process and due to this the reduction of compressive and flexural strength are also noticed [58]. Also the compounds such as C-S-H, vaterite, calcite, ettringite, thaumasite, calcium mono-carboaluminate and gypsum are detected in the twelve year old coir fibre reinforced composites and these compounds are responsible for mineralization of fibres inside the matrix [20]. A coir fibre reinforced concrete under sea water also verified the presence of high amount of ettringite and gypsum and shows saturation of both matrix and fibres with salt crystals and this suggests that both matrix and fibre degradation occurs under aggressive environment [41]. The durability improvement techniques adopted to natural fibre reinforced composited shows better behaviour of composites and this is evaluated by XRD and thermal analysis. From this evaluation, it is proved that the TMP fibre composite doesn't shows any difference in C-S-H and CH on fibre surface before and after exposure to wet/dry cycles and this suggest that there is no evidence of fibre degradation inside the composite [39]. The SCM incorporated sisal fibre reinforced composite and kraft pulp fibre composite after hot water immersion for 6 months and 25 wet/dry cycles shows reduced CH content and this is confirmed with the composite with the composite with higher strength and toughness [56, 57]. The DTA curves presented the shift of curve peak to higher temperature for treated hemp fibre with caustic soda and lime saturation solution and this shift suggests that the thermal stability is improved after fibre treatments [58]. The improvement of eucalyptus reinforced composite by early CO_2 curing shows the lower intensity of portlandite peak, which is responsible for durability of fibre but in case of non-carbonated samples higher intensity peak of portlandite is observed [54]. This is also confirmed by TGA data with higher weight loss for carbonated samples at 700^0C, showing higher $CaCO_3$ content, which leads to durability of fibres inside the carbonated matrix [54]. In addition, incorporation of metakaolin into sisal fibre cement composite after 25 wet/dry cycles shows reduction of peak of cellulose in DTA curves but this reduction is more significant in cement matrix than in metakaolin matrix and also TGA data supports this fact by showing 15% weight loss in peak of cellulose in cement matrix but only 5% loss is observed for metakaolin based matrix [40].

5. TECHNIQUES PRACTICED TO IMPROVE DURABILITY OF NATURAL FIBRES AND NFC COMPOSITES

As mentioned earlier, durability of natural fibre reinforced composites is a major concern and this fact need to be focused to overcome the degradability effects of composite exposed to different exposure conditions and to produce better products with reasonable service life. Therefore, based on the above view, there is several durability improvement techniques has been formulated till date based on various research outputs. In order to enhance the strength and durability of natural fibre reinforced cement/cementitious composites, different techniques viz. matrix modification, fibre surface modification and combined fibre and matrix modification etc. are adopted and successful results were presented in previous literatures.

5.1 Durability enhancement by fibre modification

It is well known that cement matrix itself providing unsafe environment for the degradation of reinforced fibre. From this view point, it could be a reasonable way to initially protect the fibres from the deleterious effects created by the cement matrix. Therefore, fibre surface modification and fibre structure (physical and chemical) modifications are derived as protective ways for safeguarding the natural fibres from the alkalinity effects. For such modifications, several physical and chemical methods are adopted to enhance the behaviour of fibres after incorporating into the cement matrices.

5.1.1 Surface modification of fibres

(i) Beating and bleaching

Beating is a mechanical surface treatment method and after such treatment, the fibres undergo shortening and fibrillation. On the other hand, bleaching involves removal of lignin, a polymer responsible for mineralization of fibres. The beating and bleaching treatment influences the mechanical and durability behaviour of the natural fibre reinforced composites. It is found that the fibre beating has influenced the flexural strength of the autoclaved wood fibre reinforced composite to increase and flexural toughness to reduce because of shortening and fibrillation of fibres after beating [59]. However, the bleached pulp fibre cement exhibited reduced flexural strength and toughness [60]. In terms of durability, the beaten kraft pulp fibre after 5 wet/dry cycles presented increased strength and toughness and provided better bonding as compared to that of unbeaten fibre composites. Also the beaten fibre composites exhibited improved peak strength with 10.5 % increase and post cracking toughness with 12.9% increase after 25 wet/dry cycles than that of unbeaten fibre [38]. The bleached fibre on the other hand does not show positive effect on composite durability, showing more fibre mineralization after 25 wet/dry cycles. On contrary to this, the bleaching of kraft fibre composite showed interlayer bonding effect while unbleached fibre demonstrated fibre-cement debonding during initial wet/dry cycles [39]. In an another study, softwood pulp fibre reinforced composites with unbleached, semi-bleached and fully bleached fibres shows that the fully bleached fibres presented higher thermal stability before and after subjected to 4 wet/dry cycles as compared to semi-bleached and unbleached composites [61].

(ii) Alkali treatment

The alkali treatment of natural fibre provides good efficiency in reducing water absorption capacity and modifies the surface of fibre. These changes will have beneficial effect on mechanical behaviour of the composites. Usually the alkali treatment are done using solutions of NaOH, KOH, $Ca(OH)_2$ etc. In one of the studies, it is seen that there is improvement of about 39% of flexural strength after NaOH treatment than that of untreated hemp fibres reinforced composites. Also it is noted that the treated fibre shows better interfacial bond strength but shows no effect on young’s modulus of the composite [62]. The alkalization with NaOH also shows improvement in average stress and stiffness of the hemp fibre reinforced composite as a result of removal of impurities, which is amorphous in nature. On the other hand, $Ca(OH)_2$ treatment shows only beneficial effect in average stress values [58].

(iii) Treatment using chemicals, coupling agents and additives

The literatures shows different treatments for fibre modifications with effective fibre behaviour after treatments using different chemicals, coupling agents additive, waxes etc. These treatments are beneficial in improving both strength and durability of natural fibre composites. The previous study shows that the silane treatment with alkylalkosilane, alkyltrialkoxysilane, methacryloxypropyltri-methoxysilane (MPTS) and aminopropyltri-ethoxysilane (APTS) improved the composite behaviour. It is seen that the silane treated softwood, hardwood and newsprint wood fibres presented higher compressive strength and toughness and this improvement is about 30% as compared with untreated wood fibres. The treatment with different silane contents also had improved the compression and toughness with increase in treatment levels [63, 64]. Therefore, silane treatment accompanied by pyrolysis of bagasse fibre composite exhibited decreased effect towards the water absorption and also enhanced the adhesion with the matrix and this is evident from the mode of fibre failure, showing fibre fracture after composite failure [65]. The cellulose fibre treated with MPTS and APTS presented lower water retention of composite with MPTS treated fibre and increased water retention for APTS treated fibre

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composites. Also the improvement in MOR value is noticed with APTS treated fibres [66]. The acetylation treatment with chemical such as styrene, acrylic acid, malic anhydride reduced the water absorption of fibres and styrene treatment provides reduced mass gain after water immersion and the reduction is noticed to be around 92% [67]. Sodium silicate and potassium silicate treatments are done for wood fibres and the composites with treated fibre resulted in higher compressive strength for sodium silicate and higher bending and strength for potassium silicate and normalized toughness for potassium and sodium silicate treatments. In all the cases, the treated fibre shows superior behaviour than that of untreated wood fibre cement composites [64]. Treatment of cypress particles with hot water and calcium chloride solution showed the achievement of greater strength and durability than the untreated fibre. It shows that there is no measurable degradation of boards in terms of weight loss after exposing to accelerated laboratory conditions [68]. Another chemical treatment with chloride aluminium solution presented the 7% improvement in flexural strength for treated hemp fibre reinforced composite than that of untreated case [62]. EDTA treatment plays a vital role in absorbing calcium ions on the fibre surface, thereby improving the interface between fibre and matrix for hemp fibre incorporated lime matrix [58].

Apart from chemical treatments, the natural additives are also plays significant role in enhancing the fibre properties. The additives such as linseed oil, colophony, paraffin wax, rosin etc., are believed to enhance the mechanical performances of the composite. Of all the treatments with natural compounds, paraffin wax treatment showed superior behaviour in terms of water absorption, ductility, tensile strength of fibre after exposure to 6 months in calcium hydroxide solution. Also it is reported that the paraffin treated fibre maintained about 50% of strength and ductility but other treatments showed only 12% of the initial value [36]. The surface treated sugar beet pulp with cement followed by linseed oil presented lesser water absorption and swelling behaviour, showing only little dimensional variation of the composites [69]. The heat treatment with linseed oil coating also proved to show higher tensile strength, compactness and lesser water absorption when compared to untreated diss fibre reinforced composites [63].

The polymers are also identified to better serve in modifying and mitigating the degradation of natural fibres. In this regard, the acrylic emulsion treatment of wood fibres (hardwood, softwood and newsprint) reinforced cement composites showed high average bending strength, toughness and compressive strength than that of untreated wood fibre composites [63]. Moreover, latex and resin type coating are compared for hemp fibre reinforced pozzolanic matrix and this show that the resin coating is better than latex coating in terms of flexural strength of the composite [70].

5.1.2 Pulping of fibres

Pulping of natural fibres is another technique to improve the fibre properties and makes it sustainable under degrading environment. The pulping removes the lignin (a material causing degradation effect) in greater extent and provides the good mechanical behaviour for the composites with pulped fibre. There are two different pulping usually practiced namely mechanical and chemical pulping. In mechanical pulping, usually heat application is responsible for pulping the fibres but for latter case, soaking of fibres in chemical medium is adopted for pulping process and this chemical pulping is often referred to as kraft pulping. From the study, it is noticed that the sisal pulp reinforced cement mortar performed well under flexure than that of sisal strands [71]. Also thermo-mechanical pulping (TMP) and chemi-mechanical pulping (CTMP) are adopted for sisal fibre and the cement composites with CTMP fibre exhibited reduced flexural and greater toughness than TMP fibre [39]. In terms of durability, the composites with kraft pulp and TMP after wet/dry cycles were compared. From this, it is seen that the bleached TMP fibres provides no debonding of fibres from matrix and also found to minimize the fibre mineralization than that of bleached kraft fibre composites [39].

5.2 Durability enhancement by matrix modification

Matrix modification is achieved by replacing the OPC with SCM's such as fly ash, silica fume, GGBFS, metakaolin etc, in the matrix as blends. Also other natural additives are also preferred for matrix modification namely colophony, tanin, montan wax, paraffin wax etc. Moreover, some special cement such as low alkaline cements is also used to enhance the durability of natural fibre composites. The matrix modification by SCM's are tried with blends namely binary, ternary and quaternary blends and more number of blending achieved better performance than replacing a particular type of pozzolan. Therefore binary blends of silica fume, GGBS, flyash, metakaolin proved to be very effective means of achieving durability of composites under various exposures medium. The embrittlement of sisal fibre concrete under extreme exposure mediums is found to be completely avoided by blending 40% silica fume with that of OPC. This improvement of matrix behaviour is due to reduced alkalinity of pore water present inside the concrete [1]. Also silica fume blending improved the sisal fibre reinforced mortar roofing tiles after exposure to hot weather climates even after 730 days of exposure. It is evident from the study that the OPC composites showed embrittlement of fibre within 180 days exposure and also lost all its toughness but the replacement with silica fume achieved ductility after 180, 365 and even 730 days of exposure [43]. Even under repeated wet/dry cycles, the softwood kraft fibre composites with 30% replacement of silica fume showed positive behaviour in controlling degradation and moisture effects on composites. The flexural strength and toughness performances are better than flexural strength of the composites [6]. Incorporation of blast furnace slag (BFS) into Eucaliptus grandis pulp, sisal pulp and banana pulp reinforced composite shows the good condition of fibres even after one year natural weathering. This is also confirmed by fibre-matrix interface and improvement of toughness for pulp fibres in BFS matrix and this is shown in Fig.4 [44]. Also SEM images in Fig.5 showed the pullout of fibres from the composites and the mechanical improvement is observed as the result of BFS incorporation made the matrix with high compactness and reduced capillary permeability [43]. The binary blends of silica fume, GGBS, fly ash and metakaolin are used as OPC replacements in sisal, coir and fique fibre reinforced mortar composites. This shows that the 30% improvement in compressive strength, greater reductions of capillary pores, reduced chloride penetration and better behaviour under electrochemical evaluation for the pozzolanic composites. As overall, the incorporation of silica fume showed superior behaviour and fly ash exhibited least behaviour compared to all other pozzolanic composites [73]. In addition to the binary blends, ternary and quaternary blends of SCM's are adopted to improve the durability and mechanical behaviour of the composites and their data is provided in Table 2 and 3. It is seen that the kraft pulp reinforced cement composites after 25 wet/dry cycles shows good flexural strength and toughness for all binary, ternary and quaternary blends of silica fume, slag, fly ash, metakaolin and volcanic ash. This improvement is explained by the reduction of calcium hydroxide content and ettringite reprecipitation on fibres and matrix due to supplementary C-S-H, monosulphate and calcium aluminate hydrate (C-A-H) provided by the pozzolanic activities of SCM's [56]. Also the cellulose fibre pulp with ternary blends of cement gypsum and fly ash are significantly supported to retain the mechanical performance after 100 cycles of soak/dry accelerated aging but 100% cement matrices showed adverse mechanical behaviour [74]. The other approaches tried for reducing alkalinity of cement matrices by using pumice powder, gypsum, high alumina cement and low calcium hydroxide cement. These materials used as replacements for cement shows favourable effect in enhancing the durability of the composites. It is seen that the high alumina cement, pumice powder and gypsum reduced the alkalinity of matrix reinforced with sisal fibre and also tuned the performance of matrix after exposure to aggressive environment [1, 43]. The use of special cement i.e. low calcium hydroxide cement by incorporating soft wood kraft fibre achieved the flexural stiffness and toughness after exposed to repeated wet/dry, freezing/thawing and hot water bath and these performances are better than 100% OPC composite [6]. Some of natural organic additives such as colophony, tanin, montan wax, linseed oil etc are also utilised as improvement techniques in mitigating the degradation effects by matrix modification. It is noticed from the colophony and tannin resulted in reduction of pH from 12 to 9 and this protects the fibres against alkaline degradation [22]. Moreover, the application of various durability improvement techniques such as fibre and matrix modification in separate aspects, combined fibre – matrix modification provides better way for durable natural fibre reinforced cement composites. Keeping this view point as a main factor, it is studied that using of colophony, tanin and montan wax for both matrix and fibre modification of sisal fibre reinforced cement mortar served better in different ageing conditions of continuous immersion in water and alternate wet/dry cycling. The results shows the colophony is effective than other compounds in reducing mineralisation and also the combined treatment (fibre + matrix modification) are found to increase the durability of composites after subjected to accelerated ageing [22]. In another case, the sisal and coir fibre composite is produced by modifying matrix with silica fume and BFS and fibre with silica fume slurry. The composite behaviour under flexure is observed to retain their toughness and strength almost equal to control specimens even after 322 days of outdoor weathering as well as alternate wet/ dry conditions. This effect is mainly due to the fibre immersion with binary blends of slag and silica fume [49]. The agave lecheguilla fibre treated with colophony, paraffin wax and linseed oil and matrix modification with fly ash also found to enhance the flexural strength after exposure to wet/dry cycles, chloride and sulphate solutions than that of unmodified fibre reinforced composite. Among all treatments paraffin treatment showed superior behaviour under all exposure conditions [36]. Polymers are also adopted in modifying matrix as well as fibres for reducing mineralisation of fibres under alkaline matrix. The latex and resin coated hemp fibre composite durability and this is evident from the evaluation of matrix under three point bend test [70].

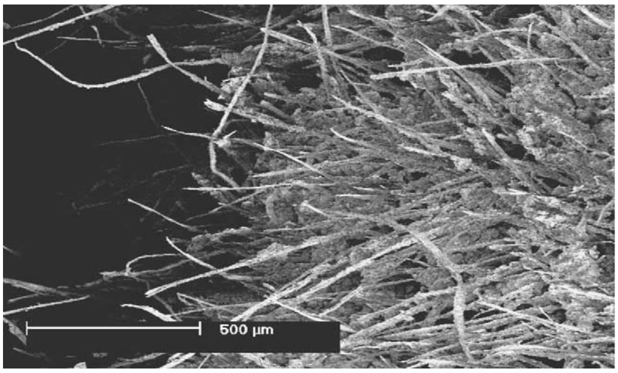

Figure-4. Fracture surface of banana kraft fibre in BFS after 12 months exposure to hot climate [44].

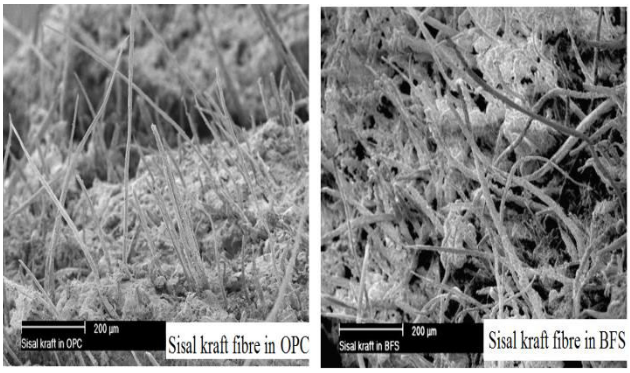

Figure-5. Good condition of kraft sisal fibre in OPC and BFS composites [72].

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Table-2. Post-cracking toughness (MPa mm) of ternary and quaternary SCM kraft fiber-cement composites [56]

	Number of wet/dry cycles													
Sample characteristics	0		1		2		5		10		15		25	
Control	8.57	(2.15)	3.29	(0.24)	0.69	(0.16)	0.17	(0.09)	0.10	(0.04)	0.11	(0.05)	0.11	(0.06)
10% SF/30% CA	4.58	(0.22)	6.11	(2.04)	4.02	(1.48)	2.39	(0.57)	0.70	(0.10)	2.76	(0.16)	0.36	(0.22)
10% SF/30% FA	3.93	(0.69)	5.03	(1.10)	4.78	(1.11)	1.95	(0.39)	0.33	(0.07)	0.20	(0.06)	0.21	(0.05)
10% SF/30% SL	8.22	(0.42)	8.13	(0.64)	5.91	(1.44)	2.10	(0.93)	0.74	(0.26)	0.38	(0.06)	0.47	(0.15)
10% SF/50% SL	5.58	(0.76)	6.67	(1.75)	5.71	(0.93)	5.30	(1.96)	2.42	(0.51)	1.52	(0.26)	1.11	(0.20)
10% SF/70% SL	6.54	(0.39)	7.38	(0.86)	6.84	(2.36)	6.34	(0.95)	6.26	(0.92)	6.12	(1.23)	5.62	(0.50)
10% SF/10% MK235	5.19	(0.86)	6.28	(1.49)	6.69	(0.76)	1.61	(0.57)	0.41	(0.04)	0.26	(0.08)	0.17	(0.01)
10% SF/10% MK349	5.65	(1.62)	5.31	(1.15)	4.30	(0.40)	1.45	(0.12)	0.61	(0.10)	0.21	(0.08)	0.15	(0.01)
10% MK235/30% SL	6.00	(1.71)	5.62	(0.78)	4.81	(0.65)	0.82	(0.15)	0.42	(0.16)	0.25	(0.06)	0.26	(0.01)
10% MK235/70% SL	5.35	(1.03)	7.99	(2.19)	6.95	(1.86)	6.99	(0.79)	5.84	(1.44)	3.28	(0.59)	2.58	(0.59)
10% CA/30% SL	9.16	(1.42)	5.34	(0.65)	3.02	(0.60)	1.09	(0.38)	0.24	(0.04)	0.13	(0.07)	0.27	(0.20)
10% CA/50% SL	6.65	(1.01)	5.10	(1.11)	3.25	(0.66)	0.73	(0.12)	0.32	(0.07)	0.32	(0.12)	0.29	(0.06)
30% CA/30% SL	7.29	(1.45)	6.81	(1.74)	3.47	(0.83)	0.54	(0.16)	0.59	(0.23)	0.36	(0.17)	0.46	(0.06)
10% FA/30% SL	8.42	(1.64)	5.68	(0.80)	2.90	(0.66)	0.66	(0.15)	0.17	(0.02)	0.21	(0.04)	0.15	(0.04)
10% FA/50% SL	5.09	(1.96)	6.93	(1.58)	2.20	(1.18)	0.87	(0.20)	2.57	(0.13)	0.34	(0.05)	0.37	(0.18)
30% FA/30% SL	5.27	(1.06)	4.71	(0.60)	2.61	(0.22)	0.47	(0.10)	0.40	(0.22)	0.42	(0.02)	0.17	(0.06)
10% MK235/10% SF/30% SL	7.32	(1.76)	7.02	(2.16)	6.18	(1.26)	3.29	(0.26)	1.30	(0.30)	1.01	(0.34)	1.10	(0.23)
10% MK235/10% SF/50% SL	6.32	(1.81)	7.60	(1.35)	10.68	(2.51)	8.87	(0.74)	5.48	(1.14)	4.28	(0.64)	2.67	(0.50)
10% MK235/10% SF/70% SL	7.10	(0.33)	8.42	(2.50)	8.27	(1.70)	7.97	(1.49)	8.48	(1.12)	7.61	(1.55)	8.28	(2.57)

Table-3. Peak strength (MPa) of ternary and quaternary SCM kraft pulp fiber-cement composites [56].

	Number of wet/dry cycles													
Sample characteristics	0		1		2		5		10		15		25	
Control	10.34	(0.36)	5.80	(1.03)	3.83	(0.13)	2.25	(0.72)	2.63	(0.26)	2.83	(0.33)	2.85	(0.59)
10% SF/30% CA	8.04	(0.65)	7.96	(1.08)	6.66	(0.90)	5.65	(0.70)	3.10	(0.32)	2.62	(0.27)	2.50	(0.64)
10% SF/30% FA	7.22	(1.17)	7.26	(0.70)	7.24	(0.80)	5.01	(0.71)	3.10	(0.28)	2.49	(0.14)	2.33	(0.39)
10% SF/30% SL	9.82	(0.84)	7.45	(0.84)	6.92	(1.55)	4.42	(0.36)	2.88	(0.17)	2.02	(0.23)	2.14	(0.67)
10% SF/50% SL	11.74	(0.66)	9.44	(0.70)	8.41	(1.04)	8.19	(1.33)	6.06	(0.59)	4.68	(0.86)	3.95	(0.50)
10% SF/70% SL	11.91	(0.95)	9.29	(0.47)	8.39	(1.14)	8.60	(0.61)	7.72	(0.80)	7.67	(0.78)	6.47	(0.33)
10% SF/10% MK235	9.46	(0.89)	9.46	(2.20)	9.40	(1.25)	5.79	(1.17)	2.98	(0.41)	2.69	(0.06)	2.25	(0.42)
10% SF/10% MK349	9.38	(1.32)	7.80	(0.49)	7.49	(0.37)	4.36	(0.25)	3.41	(0.38)	2.22	(0.43)	2.67	(0.43)
10% MK235/30% SL	8.84	(1.75)	7.94	(0.56)	7.74	(0.82)	3.44	(0.27)	2.01	(0.40)	1.54	(0.05)	1.27	(0.33)
10% MK235/70% SL	8.12	(0.74)	7.67	(0.36)	7.44	(0.32)	8.26	(0.02)	9.01	(0.84)	6.78	(0.72)	6.01	(0.72)
10% CA/30% SL	9.68	(1.24)	6.65	(0.43)	5.86	(0.61)	3.46	(0.35)	2.52	(0.35)	1.68	(0.39)	1.74	(0.22)
10% CA/50% SL	9.58	(0.53)	8.00	(0.72)	6.48	(0.83)	3.66	(0.19)	2.51	(0.47)	1.94	(0.72)	2.48	(0.13)
30% CA/30% SL	5.89	(0.82)	5.55	(0.83)	4.90	(0.84)	2.49	(0.16)	2.44	(0.35)	2.19	(0.23)	2.07	(0.47)
10% FA/30% SL	9.52	(1.16)	6.20	(0.49)	5.60	(0.54)	2.99	(0.21)	2.22	(0.58)	1.95	(0.19)	1.41	(0.21)
10% FA/50% SL	9.53	(1.95)	7.40	(0.90)	6.02	(1.40)	3.04	(0.54)	1.98	(0.16)	2.31	(0.30)	1.92	(0.26)
30% FA/30% SL	6.88	(0.87)	6.34	(0.42)	6.03	(0.59)	3.22	(0.66)	1.70	(0.27)	2.06	(0.04)	1.54	(0.02)
10% MK235/10% SF/30% SL	10.54	(0.94)	8.26	(1.31)	7.99	(0.95)	6.44	(1.12)	3.70	(0.26)	3.00	(0.36)	3.07	(0.22)
10% MK235/10% SF/50% SL	9.49	(0.59)	8.75	(0.40)	11.51	(2.05)	10.48	(0.47)	9.21	(1.86)	7.94	(0.89)	5.72	(0.20)
10% MK235/10% SF/70% SL	8.16	(0.42)	7.31	(0.45)	7.09	(0.49)	8.12	(0.84)	7.97	(1.32)	7.87	(0.50)	7.99	(0.86)

5.3 Improvement of durability of NFC composites by CO_2 curing

Early CO_2 curing of natural fibre cement composites is found to increase the durability and engineering properties. It is seen that increased carbonation effects causing the wheat straw cement board to retain the desirable flexural strength and bond strength properties even after exposure to wet/dry cycles, freeze/thaw cycles and warm water immersion. Also the micrographs presented the good intact of wheat straw with cement board with limited micro-cracks [51]. The cellulose fibre composites also exhibited better flexural stiffness and reduced capillary porosity of composites and improved bonding due to increased $CaCO_3$ content after subjecting to various aggressive exposure mediums [75]. The denser $CaCO_3$ leads to better fibre-matrix bond and improved mechanical behaviour for the eucalyptus cellulosic pulp reinforced cement composite after exposure to accelerated and natural ageing, showing mitigation of composite degradation [54]. The CO_2 cured pressed and unpressed cellulose fibre cement composites showed reduced water absorption than non carbonated composites. Also flexural stiffness and strength are improved and toughness is reduced for CO_2 cured composites than that of normal cured composites [76].

6. CONCLUSIONS

Natural fibre reinforced cement/cementitious composites have prospective future in the field of civil engineering applications. The various natural fibres such as sisal, malwa, eucalyptus, coir, banana, hemp, wood fibres etc. are widely used potentially for various applications in low cost housing. The various ill effects associated with natural fibres due to water affinity and degradation under alkaline solutions causes little interest on natural fibres composites to be used for longer service life under extreme climatic conditions. From the extensive literature review, it is concluded that there are several improvement techniques adopted for enhancing the durability of various natural fibre reinforced composites. The improvement techniques so far practiced really shows promising results and better end products even after exposure to aggressive medium both in laboratory and natural weathering conditions. Adopting these techniques in combined aspects for producing particular composites resulted in good mechanical performance and this shows that the natural fibre cement products have been found to be a best option for synthetic fibrous composites and also open a wide market for various civil engineering applications.

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