



FLEXURAL CHARACTERIZATION OF POLYMER CONCRETE COMPRISING WASTE MARBLE AND DATE PALM FIBERS

Mansour Rokbi¹, Zine El Abidine Rahmouni², Brahim Baali³

¹Department of Mechanical Engineering
Faculty of Technology

University of M'sila, Algeria

²Department of Civil Engineering
Faculty of Technology

University of M'sila, Algeria

³Sarl Maghreb Pipe
Industriel Zone, M'sila, Algeria

URMPE/MESOnexTeam

Faculty of Engineering
University of Bumerdes, Algeria

Received 7 April 2019, accepted 29 May 2019, available online 3 June 2019.

Keywords: Polymer concrete, Date palm fibers, Quartz, Marble, Fibers sizing, Three-point bending.

Abstract

This work is an experimental approach for the development and characterization of a polymer concrete reinforced with natural fibers. The polymer concrete consists of sand (Quartz) and orthophthalic polyester used as a binder. Marble powder was used to ensure the continuity of the particle size of the granular mixture. As reinforcement, 2% of chopped date palm fibers (short, very short or mixed) were added. For comparison, identical polymer concrete flexure specimens reinforced with the same content of short E-glass fibers were also prepared and tested. All specimens were initially cured at room temperature and then post-cured for 6 h at 70°C. The results of three-point bending on smooth specimens with different rates of charges (marble), showed that the flexural and compressive strength were improved by adding 20% of marble, and were 31.80 MPa and 67.42 MPa respectively. The flexural strength of specimens showed that the improvement or the degradation of polymer concrete properties seemed to be attributed to the nature of fibers (treated or untreated), and/or to the fibers sizing (short, very short or mixed).

Correspondence: Mansour Rokbi, Department of Mechanical Engineering, Faculty of Technology, University of M'sila, 28000, Algeria, phone: +6 99 03 88 05, e-mail: mansour.rokbi@univ-msila.dz

Introduction

Polymer concrete (PC) is a material composed of different types of aggregates bound together by polymeric resins sometimes thermoplastic but generally thermosetting. The lightness of this material and its water absorption percentage practically nil guarantee a total waterproofness of the material, moreover its inalterability to the cycles of freeze-thaw, its high resistance to most chemicals and impact, as well as its minimal wear by abrasion are other features that make polymer concrete a high quality material, and therefore, long life of concrete structures can be considered (MOHAMMADHOSSEINI et al. 2017). Compared with Portland cement concrete, polymer concrete has greater mechanical strength, Knowledge of the behavior of these materials and its development is necessary. In the context of sustainable development, intense efforts are made by researchers to develop new alternative materials including recycled materials or integrating industrial waste. This initiative focuses on producing health-friendly materials by reducing the environmental impact (MAGNIONT et al. 2012, ROKBI et al. 2017). In other words, many waste materials from various industrial sources can be seen as potentially valuable materials (AWAL, MOHAMMADHOSSEINI 2016). Naturally available materials such as waste marble which is an environmental problem, can be used effectively as mineral aggregate (ROKBI et al. 2017).

Recently, several works deal with the reinforcement of polymer concrete. The most used reinforcements are glass fibers, steel, polypropylene (PP), waste fiber and natural fibers (AWAL, MOHAMMADHOSSEINI 2016, MOHAMMADHOSSEINI et al. 2018). Nowadays, the reinforcement of concrete structures by natural fibers has attracted substantial interest, and there are various research regarding this particular topic in related literature (ROKBI et al. 2017, BOUGUESSIR et al. 2018, REIS 2012). The advantage of incorporation of natural fiber in polymer concrete structures is because these fibers are abundantly available from renewable resources, biodegradable, renewable and have low density and high specific properties. In addition, the availability of natural fibers in many underdeveloped countries such as Brazil, India and Bangladesh allows them to develop low-cost construction materials with little technology support and a small amount of energy. Lignocellulosic fibers have been used as a reinforcing agent in conventional concrete as well as in innovative concrete. The main purpose of these investigations is to replace metallic or synthetic fibers .

To analyze the possibility of substitution of synthetic fibers by natural fibers, REIS (2006) investigated the mechanical characterization (flexural strength, fracture toughness and fracture energy) of epoxy polymer concrete reinforced with different chopped natural fibers (coconut, sugarcane bagasse and banana fibers). Careful analysis of these results shows that some of these fibers (Coconut and sugar cane bagasse) proved to be an alternative when fracture properties are analyzed. In return, the low interfacial properties between lignocellulosic

fibers and an organic resin system (or inorganic matrix) often reduce their potential as reinforcing agents due to the hydrophilic nature of natural fibers, chemical modifications are considered to optimize this disadvantage. To improve the interfacial characteristics between lignocellulosic fibers and the matrix, several surface modifications including alkali, coupling agents and acetylation have been investigated. The treatment with sodium hydroxide (NaOH) is relatively simple and widely being used to modify the cellulosic structure like Alfa, jute, sisal, kenaf, palm or banana (ROKBI et al. 2018). The experiment work of ACHOUR et al. (2017) show that the treatment of lignocellulosic fibers such as Diss and Doum with NaOH solution (3% and 1% respectively) are very effective to improve adhesion between natural fibers and inorganic matrix. Once treated, the lignocellulosic fibers can contribute to a good interaction between fiber and the cementitious matrix by limiting the propagation of microcracks, by a sewing effect; which improve the performance of cementitious mortars. In recent work (REIS 2012), fiber surface treatments were carried out to enhance bond interface between sisal fiber and polymer matrices to improve the fracture properties. In this study, the effects of the incorporation of the untreated and treated sisal fibers with NaOH and acetic acid into two polymeric mortars, based on epoxy and polyester resins respectively were investigated. Untreated and surface treated sisal fibers when used as reinforcement contributes significantly to improve the fracture properties both energy and toughness of epoxy and unsaturated polyester polymer mortars. It has also been observed that polymer mortars reinforced with untreated sisal fibers have the highest ultimate strength and those reinforced with sisal fibers treatment with 10% of NaOH have the lowest properties. ROKBI et al. (2017) have study the feasibility of using naturally available materials: Alfa fibers and waste marble powder (WMP) in PC material. In their study, two weight percentages of Alfa fibers (1% and 2%) were used. The results show that the reinforcement with 1% of Alfa fiber can improve the fracture toughness better than 2% fiber reinforcement. In addition, the quality of the adhesion between the natural fiber and polymeric resin appeared good when Alfa fiber was treated with 5% NaOH.

It is known that during the process of cross linking of unsaturated polyesters shrinkage and cracking phenomena are quite present and represent major problems. In addition, this type of resin is not hard enough and has a lower impact strength compared to other thermoset resins such as epoxy. To overcome these problems, several researchers have proposed the use of many types of fillers such as calcium carbonate, silica, alumina trihydrate, clay, mica, fly ash, etc. Thus, the properties of the resin and the filler, the dispersion of the particles, and the state of the interface resin play a primordial role on the mechanical properties of the PC (HRISTOVA, MINSTER 2003, ANISKEVICH, HRISTOVA 2000, VYTLACILOVA 2011, CHOUDHARY et al. 2019). HRISTOVA and MINSTER (2003) described the effect of a particle marble filler on the creep response of a cross-linked

polyester matrix before and after physical aging. It was observed that the values of the creep compliance decrease and the values of the modulus of elasticity increase with increasing filler volume fraction.

The purpose of the present work was to discuss the usability of the naturally available materials: waste marble powder (WMP) and date palm fruit bunches fibers (DPF) in PC material. Firstly, the optimum of the amount of WMP addition by weight was identified and reported. Secondly, the effects of fruit bunches fibers sizing (short, very short or mixed) as well their treatment (5% NaOH during 24 h) as reinforcement on the mechanical performance of PC have been studied.

Materials and Methods

Materials

Fiber and resin

In this study, the date palm fruit bunches fibers were used as reinforcement in PC. These fibers were collected from the region of Biskra. It is a semi-arid region in Algeria. Once the fruit bunches were harvested, they were washed with water (2% detergent solution) to remove the contaminants and adhering dirt. Thereafter, fruit bunches were cut into 6cm and 2cm lengths (Fig. 1a).

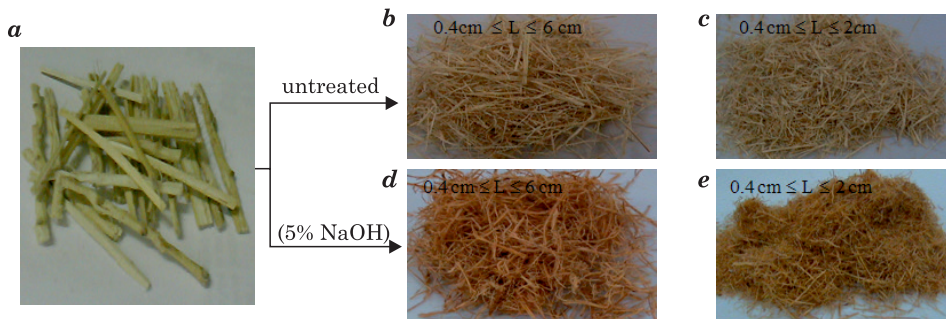


Fig. 1. Different form of used fibers: *a* – DPF before extraction, *b* – Untreated (short), *c* – Untreated (very short), *d* – Treated (short), *e* – Treated (very short)

Pretreatment techniques and fibers processing are similar to those described in (ROKBI et al. 2011). The chopped fruit bunches were soaked in a 5% NaOH solution at 28°C. The fibers were kept immersed in the alkali solution for 24 h. The treated fruit bunches were then washed several times with distilled water. Any traces of NaOH, remaining on the fiber surface, were neutralized with

2% sulfuric acid during 10 min. The fibers were washed again with distilled water until obtaining a pH = 7. Subsequently, the fibers were dried at 60°C for 6 hours. Untreated and treated fibers are represented in Figure 1*b*, *c*, *d*, and *e*. Like other plant fibers, date palm fruit bunches fibers are characterized by good mechanical performances (strength = 397,2 MPa, Young Module = 6.9 GPa) (TAHRI et al. 2016).

The used resin is orthophthalic polyester. It is a pre-accelerated resin with good mechanical properties. It is specially designed for laminating and hand lay-up technique.

Mineral fine aggregate

The aggregate used in PC was Quartz fine sand with a homogeneous grain size with an average diameter of 200 to 500 μm . The chemical compositions of the used Quartz fine sand are listed in Table 1.

Table 1

Chemical compositions of Quartz sand							
Fine sand [%]	MgO	CaO	Fe ₂ O ₃	Al ₂ O ₃	SiO ₂	TiO ₂	H ₂ O
	0.006	0.010	0.215	0.769	99.02	0.078	0.02

Mineral addition

In this work very fine WMP has been used in the PC as a mineral addition. Marble powder is a compact metamorphic rock marketed through the manufacture of ceramic tiles from an industry in Fil-Fila quarry (Skikda region in the north-east of Algeria). WMP is a useful material obtained as a by-product of marble during sawing, shaping, and polishing process such that about 25% of the processed marble turns into dust or powder form (GÜNEYİSİ et al. 2009). The recovered waste marble is sieved into marble powder using a fine sieve. The characteristics of marble powder are presented in Table 1 and 2.

Table 2

Physical characteristics of waste marble powder	
Couleur	White
Structure	Micro-crystalline
Specific gravity	2.68 g/cm ³
Blaine fineness	7.50 cm ² /g

Table 3

Chemical composition of marble powder									
Oxides	SiO ₂	CaO	MgO	Fe ₂ O ₃	Al ₂ O ₃	Na ₂ O	K ₂ O	SO ₃	LOI*
%	1.47	55.30	0.01	0.14	0.35	0.12	0.04	0.01	42.56

*LOI: Loss on ignition at 1000°C

In order to identify the optimum of the amount of WMP addition, flexural specimens were prepared by mixing Quartz fine sand with different WMP contents of 0%, 3%, 5%, 10%, 20% and 30% in weight (Tab. 4). For the formulation, the amount of orthophthalic polyester was taken as 20%, in mass, of concrete aggregate weight (Quartz sand and WMP) (REIS 2006). At least, five flexural specimens, for each amount of WMP, were compacted in a steel mold of dimensions of 40 × 40 × 160 mm (Fig. 3a). PC specimens were initially cured at room temperature and then post-cured for 6 h at 70°C.

The gradation of both Quartz and WMP are shown in Figure 2. The particle size distribution of WMP was measured by laser particle analyzer.

Table 4

Different manufacturing unreinforced PC						
Unreinforced PC						
Quartz sand content	100%	97%	95%	90%	80%	70%
WMP content	00%	03%	05%	10%	20%	30%
Designation	PC100-00	PC97-03	PC95-05	PC90-10	PC80-20	PC70-30

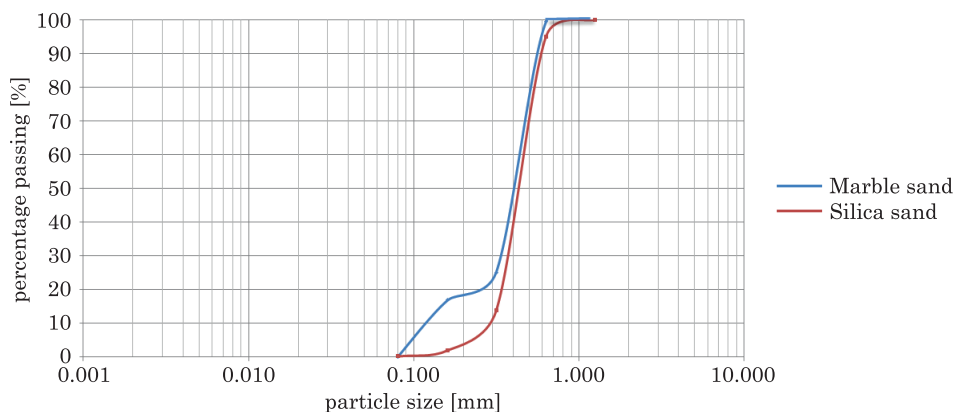


Fig. 2. Particle size distribution of Quartz and WMP

In each series, chopped date palm fruit bunches fibers at 2% of the total weight of specimen was used according to (REIS 2006). Based on the optimum of WMP content, eight mixtures of flexural specimens were prepared using different reinforcement (Tab. 5). As indicated by this table, and in addition to the two series of unreinforced PC specimens and those reinforced by glass fibers, two groups of specimens are elaborated in this investigation: the first group consists of three series of PC specimens reinforced by untreated fibers which are respectively PC/very short fibers (PC-A), PC/short fibers (PC-B) and PC/mixture of fibers (PC-AB). In a similar way, the second group consists of the same formulation, but in this case the three series of PC specimens are reinforced respectively by treated fibers which are PC-C, PC-C and PC-CD.

Table 5

Different manufactured PC			
PC	Fibers sizing	Max Fiber length [mm]	Designation
PC /Unteated DPF Group 1	very short	2	PC-A
	short	6	PC-B
	mixed (50% V. Short+50% Short)	6	PC-AB
PC /Teated DPF Group 2	very short	2	PC-C
	short	6	PC-D
	mixed (50% V. Short+50% Short)	6	PC-CD
Unreinforced PC	no fibers	/	PC-E
PC /Glass fibers	short	6	PC-F

Testing procedures

As known, both flexural and compressive properties of construction materials are very useful especially if their intended applications are country road or pavement (PEREIRA et al. 2015). In this work, the flexural and compressive tests were performed on the different PC samples. For flexure testing, the three-point bending tests were performed in a mechanical testing machine YL universal testing machines/25kN (Figure 3(b)), at a crosshead movement rate of 1 mm/min, according to the Technical committee TC-113 test methods for concrete–polymer composites (RILEM 1995). Despite the very short span length compared with specimen thickness, support span of 100 mm, shear effect is disregarded and it is not considered (REIS 2006). Flexural strength, considered as the strength under normal stresses, was determined by applying the following equation known from the strength of materials:

$$\sigma_f = \frac{3 Pl}{2 b h^2} \quad (1)$$

where:

σ_f – the flexural strength,

P – the maximum load recorded,

l – the span length,

b and h are, respectively, the width and the height of the prismatic specimens.

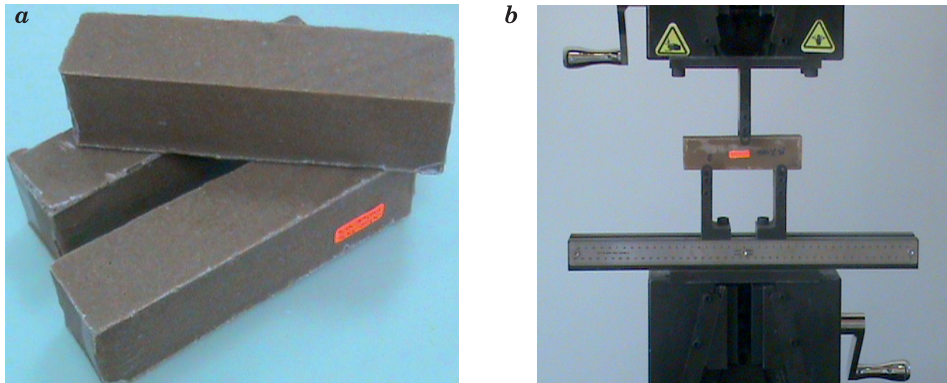


Fig. 3. Specimens and testing: *a* – 40×40×160 mm specimens, *b* – Flexural test set-up

The compression test consists of studying the ability of a material to withstand a force that attempts to crush it. In our case, the compression test is performed on cubic specimens for each concrete mixture and were tested for the determination of compression strengths using STRASSEN TEST loading machine. This latter has a crushing capacity of 2000 kN. The compression load was applied at a rate of 2.4 kN/s.

Results and discussion

Effect of WMP replacement

In this paragraph, the effect of substitution of fine sand by different WMP contents on the mechanical performances of PC was studied in order to optimize the amount of WMP added as replacement of Quartz in PC. The WMP is used in various industrial applications as a filler material and it could be assumed as ultrafine aggregates filling voids in PC (ERGÜN 2011).

Table 6 presents the flexural and compressive test results of PC when the fine sand was replaced by different WMP contents. When small amounts of WMP were used, cases of PC97-03 and PC95-05 materials, the flexural strengths

Table 6

Flexural and compressive test results of PC						
PC	PC100-00	PC97-03	PC95-05	PC90-10	PC80-20	PC70-30
Maximum load [kN]	12.15 ±0.83	9.67 ±1.33	9.90±1.97	11.89±0.43	14.63±0.37	14.72±0.20
Flexural strength [MPa]	27.9 ±0.85	21.20±1.23	21,60±1.05	27.10±1.23	31.80±0.42	31.10±0.62
Flexural strain [%]	2.41±0.15	1.45±0.30	1.47±0.22	1.53±0.14	1.88±0.28	1.70±0.31

Source: ROKBI et al. (2017).

of materials were decreased of about 24% compared to the materials PC100-00 (see Table 6). The remarkable reduction in flexural strength seems related to the use of a small amount of WMP which had somehow created microscopic defects, and the materials have a greater possibility of early failure. From the same table, the flexural strength of the material PC90-10 has not undergone a remarkable change. In other hand, and compared to the materials PC100-00, the flexural strength of both materials PC80-20 and PC70-30 has increased of about 12% and 10%, respectively, This may suggest that the substitution of WMP by 20% of Quartz allows registration of the best flexural and compressive strength. The use of 20% of WMP could be considered as the optimum amount to enhance the properties of PC (ROKBI et al. 2017).

Flexural test

For all flexural specimens reinforced with treated and untreated DPF and glass fiber, 20% of WMP amount were used. Figure 4 shows the load-deflection curves for each PC mixture tested in three-point bending. The load-deflection curves were similar to those of fibers reinforced thermoset matrix materials.

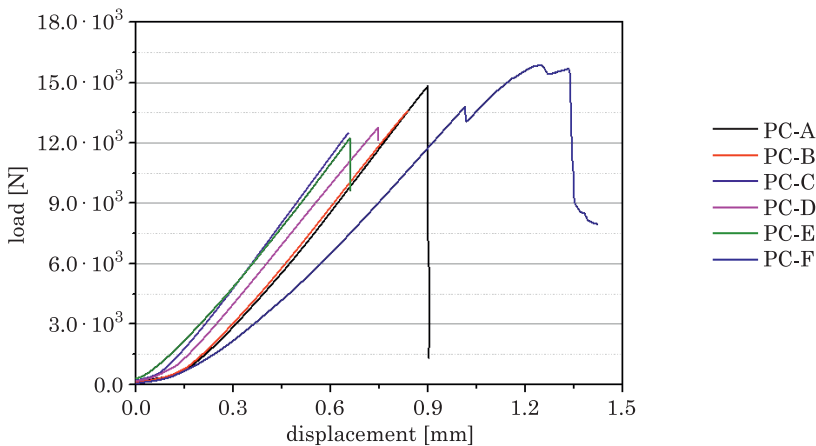


Fig. 4. Load-deflection curves of various test groups

All the tested specimens showed a brittle behavior with a linear elastic deformation up to the catastrophic failure.

From the tests, it was showed that the PC reinforced with untreated PDF fibers materials have maximum load than those reinforced with treated fibers (Fig. 4). This can be explained by the effect that the alkaline treatment can be harmful and leads to fragile fibers (ROKBI et al. 2011, CHIKOUCHE et al. 2015). NaOH concentration would certainly damage the fiber and consequently reduced the flexural strength of the PC reinforced with treated fibers.

Figure 5 shows representative histograms for flexural strength for all specimens tested under statical loading. As indicated in Figure 5, the incorporation of DPF fibers in PC had a significant effect on the flexural strength values. The use of very short untreated DPF (Mat-A) appeared more suitable for use as reinforcement in PC. In this case, an improvement in flexural strength of about 23% and 5% have been observed compared to the materials Mat-E and Mat-C, respectively. In one hand, compared to the material Mat-E, the increase in the strength of Mat-A appears to be related to the addition of fibers which play an active role as bridging mechanism by controlling resulting microcracking and reducing the crack propagation rate. In other words, the fiber reinforced concrete can transform the brittle behavior to a pseudoductile one by maintaining considerable load carrying capacity after cracking of the matrix (PEREIRA et al. 2015).

On the other hand, the remarkable improvement in the strength of Mat-A (about 23%) compared to the material Mat-C was mainly due to the fragile nature of the treated DPF fibers.

The flexural strength values in group 1 was different compared with that of group 2. The best flexural strength in group 1 was obtained when the PC was reinforced by very short fibers (case of Mat-A), while this parameter

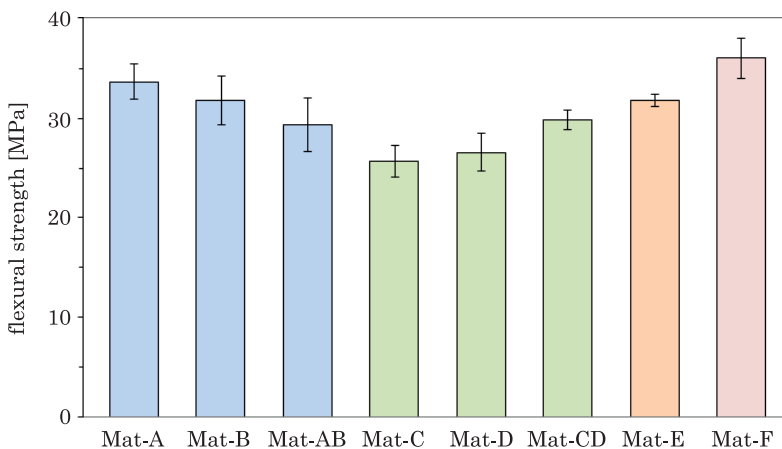


Fig. 5. The flexural strength of different PC samples

is gradually reduced once the short fibers or the mixture of fiber are used. The same observation is reported by (LI et al. 2006) when studying coir fibers reinforced cementitious composites (CFRCCs). These authors compared the flexural strength in cementitious composites reinforced by untreated short coir fiber (2 cm) to that reinforced by untreated long coir fiber (4 cm). From their results, it can be seen that some short fiber CFRCCs show better flexural strength than the long fibre (4 cm) CFRCCs. This may suggest that the long fiber is neither well dispersed nor straightened (LI et al. 2006).

Considering the flexural strength values in group 2, it is to be noted that neither the material Mat-C nor the material Mat-D have proved good results. The best strength was obtained when mixed fibers are used as reinforcement in PC (case of Mat-CD). The fragile nature of the treated fibers has favored the degradation of properties of the PC when these fibers have almost the same size. However the use of different sizes of treated fibers seemed improve the fiber bridging effect. ALSAEED et al. (2013) investigated the effect of alkali treatment of date palm fiber on its interfacial adhesion with epoxy matrix. They found that higher concentration of chemical treatment might decrease the strength of natural fiber. In other work, ROKBI et al. (2011) studied the influence of alkaline treatment on the flexural properties of polyester matrix composite reinforced with Alfa fibers. The alkali treatment, NaOH, at 1%, 5%, and 10% concentrations for various soaking periods of 0, 24 and 48 h has been conducted. The flexural strength and flexural modulus improved by 60% and 62% respectively at 10% NaOH consideration for a period of 24 h. Also, they showed that the longer treatment time of Alfa fiber (48 h with 5% NaOH) decreased the flexural strength and flexural modulus due excess delignification natural fiber that led to weakening of the fiber strength and the damage occurred on the fiber. Due to these damages, interface adhesion and wettability become poor, which decreases the composite properties.

Micrographs of Figure 6 showed the surface of the DPF fiber before and after treatment. The surface of untreated the DPF fiber was found to be considerably covered with waxy substances and impurities (Fig. 6a). Certainly, dirt and layers

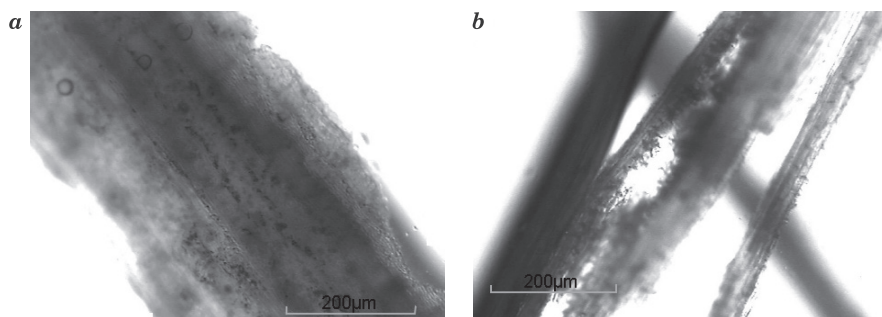


Fig. 6. Optical micrographs of surface morphology of DPF fiber: *a* – Untreated, *b* – Treated

waxy cuticle on the surface of the fibers were completely removed after NaOH treatment, but it seemed that treatment with 5% NaOH for 24 h had significantly deteriorated the PDF fibrils (Fig. 6b). At higher alkali concentration, excess delignification of natural fiber occurs resulting in a weaker or damaged fiber, and the treatment of fibers over a prolonged period makes the fibers stiffer and more brittle (ROKBI et al. 2011).

It was found that the optimum properties of DPF fibers were achieved at relatively short treatment times of 2 h and 4 h and alkali concentrations of 2% and 5% NaOH, respectively (TAHA et al. 2007).

In another work, (ALAWAR et al. 2009) investigated on the effect of alkali treatment on DPF behaviors. Their Result shows that the treatment of conditions of 1% NaOH for 1 h at 100°C is the optimum treatment that gives the maximum tensile strength and the better surface morphology of the single DPF.

Figure 7 presents the flexure fracture surfaces micrographs of the Mat-B, Mat-D, and Mat-F. The untreated fibers appear non-covered by the matrix; it was not subjected to any abrasion. The fiber pull-out with a considerable length is clearly visible (Fig. 7a). It seemed that PDF fibers needed surface treatment to ensure better adhesion. The contrary, treated DPF with 5% NaOH showed a good adhesion with aggregates. It seemed strangely that the color of these fibers became brick-red (Fig. 7b).

It appears that the treated DPF were destroyed before their introduction as reinforcement in PC. The fractured surface of the material Mat-F showed that the fiber surface of chopped glass fiber were coated with aggregate wrapping that have been partially destroyed or abraded (Fig. 7c). This may help to explain the remarkably high pull out load (BENZERZOUR et al. 2012). Compared to the Mat-A, the material Mat-F showed slightly larger maximum strength. This result suggests that local DPF are comparable to chopped glass fibers used as reinforcement in PC.



Fig. 7. Optical micrographs of fracture surfaces of: *a* – Mat-B, *b* – Mat-D, *c* – Mat-F

Conclusion

In this study, in the first step, the influence of various amount of waste marble powder on both flexural and compressive properties of the PC samples based on Quartz, waste marble powder and orthophthalic polyester were studied, and the optimum of the amount of WMP addition by weight (20%) was identified and reported.

In the second step, and based on the optimum of WMP content, eight mixtures of flexural specimens were prepared using different reinforcement for the purpose of reflecting the effect of nature of fibers (treated or untreated), and/or the fibers sizing (short, very short or mixed) on the flexural performances of PC.

The analysis of flexural results for all specimens tested under statical loading allow the following interpretations:

- the use of very short untreated date palm fruit bunches fibers appeared more suitable for use as reinforcement in PC. In this case, an increasing in flexural strength from 31.78 MPa to 33.67 MPa was attributed to the fiber bridging mechanism;

- the longer treatment time and/or higher concentration of chemical treatment reduced the performance of the natural fiber, However, the use of different sizes of treated fibers would seem to participate effectively to increase the fiber bridging effect;

- the PC reinforced with glass fibers showed slightly larger maximum strength (36.01 MPa) compared to the PC reinforced by untreated DPF (36.01 MPa). This result suggests that local DPF are comparable to chopped glass fibers, and can be used successfully as a reinforcing agent in civil engineering construction;

- finally, it is important to mention that low concentration of NaOH can give good results. Studies are in progress in this direction.

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