

Article

Enhanced Performance of Concrete Composites Comprising Waste Metalised Polypropylene Fibres Exposed to Aggressive Environments

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Abstract: The utilisation of waste plastic and polymeric-based materials remains a significant option for clean production, waste minimisation, preserving the depletion of natural resources and decreasing the emission of greenhouse gases, thereby contributing to a green environment. This study aims to investigate the resistance of concrete composites reinforced with waste metalised plastic (WMP) fibres to sulphate and acid attacks. The main test variables include visual inspection, mass loss, and residual strength, as well as the microstructural analysis of specimens exposed to aggressive environments. Two sets of concrete mixes with 100% ordinary Portland cement (OPC) and those with 20% palm oil fuel ash (POFA) were made and reinforced with WMP fibres at volume fractions of 0–1.25%. The results revealed that the addition of WMP fibres decreased the workability and water-cured compressive strength of concrete mixes. The outcomes of the study suggest that the rate of sulphate and acid attacks, in terms of mass losses, was controlled significantly by adding WMP fibres and POFA. The mutual effect of WMP fibre and POFA was detected in the improvement in the concrete's resistance to sulphate and acid attacks by the reduction in crack formation, spalling, and strength losses. Microstructural analysis conducted on the test specimens elucidates the potential use of POFA in improving the performance of concrete in aggressive environments.

Keywords: concrete composites; waste metalised polypropylene fibres; durability; sulphate and acid attacks; palm oil fuel ash

1. Introduction

The management of several types of waste has become a challenge owing to the vast quantities of waste materials produced worldwide. Cleaner productions can be achieved by minimising the generation of industrial and domestic solid wastes and the reduction in the consumption of raw resources [1]. According to Prem et al. [2], several kinds of non-biodegradable solid wastes such as plastic wastes will remain for hundreds or thousands of years in the environment. The aspiration to attain a green society is commonplace, regardless of the lack of appropriate waste management systems in both developed and developing countries. Although there is room for enhancement in existing practices, the transition to green production needs the identification of context-specific leverage points that can effect change, particularly for the informal divisions. Abdel-Gawwad et al. [3] stated that the utilisation and recycling of the waste materials generated are the key issues of waste management



considerations. Moreover, as the idea of cradle-to-cradle is of growing significance nowadays for a green society, sectors such as concrete construction must plan to reuse solid waste instead of raw materials. Consequently, green construction with lesser cost will be introduced, as pointed out by Evi Aprianti [4] and Jittin et al. [5].

The manufacture of several types of plastics has grown-up enormously globally. According to Gu and Ozbakkaloglu [6], the global manufacture of plastics in various forms rose to approximately 300 million tons in 2014. Among the total plastic waste generated, approximately half of the plastics made were used only once, which began the generation and disposal of a massive amount of waste plastics, as pointed out by Aryan et al. [7]. Accordingly, inadequate controlling and mismanagement of waste materials result in harmful effects on human life as well as the environment. Nonetheless, the mainstream of plastic wastes can be reused and reprocessed thermally or chemically, although not all sorts of waste plastics, as reported by Longo et al. [8]. Metalised plastic film wastes are generated and sent to landfills worldwide. These are mostly polymer-based and covered with a thin layer of aluminium; they are used primarily in foodstuff packing. Among all plastic wastes, WMP is unsuitable for reprocessing and recycling [9]. As an appropriate method for the recycling of such a widespread amount of waste plastics does not exist, they are consequently sent to a landfill to be burnt [10]. Consequently, reliable approaches to disposal that can replace the existing methods have become essential. Besides, POFA is an industrial waste containing pozzolanic materials. POFA is attained by burning palm fruits in a palm oil mill as fuel. As specified by Khankhaje et al. [11], in Malaysia, which is the second producer of the palm oil products in the world, in 2010, approximately 4 million tons of POFA was generated and sent to landfill. The waste POFA ashes generated from mills are now categorised as a good pozzolanic material with suitable features, which have potential to be used as cementing materials in concrete to improve the performance of concrete, as pointed out by Juenger and Siddique [12].

Generally, due to the availability of raw materials, ease of production, adequate durability and strength properties, concrete is a well-known construction material globally [13]. Nevertheless, owing to the wide applications of concrete, particularly in the industrial sectors, it is frequently exposed to chemical attacks. These attacks can be natural, e.g., by chemicals such as sulphates in soils, groundwater and seawaters, or chemical attacks made by a humans, for example, drainage wastewater and industrial sewage, which destructively impact the performance of concrete structures and shorten their service life. In this regard, Brown and Badger [14] stated that the deterioration of concrete might occur by chemical reactions owing to the exchange of ions under the impact of such aggressive environments. Consequently, the results of these attacks are in the form of degradation in the hydration products as we as the formation of harmful products such as gypsum and ettringite in the matrix. It, therefore, develops the cracks, spalling, and disparities in the microstructure of concrete and, finally, reduce the strength and durability of concrete structures.

According to Santhanam et al. [15] and Bulatovic et al. [16], sulphate particles in the solid form do not attack the concrete severely; nevertheless, with the existence of water, these sulphate particles find a way to enter into the cavities in concrete components and chemically react with the hydration products of cement. Sotiriadis et al. [17] also reported that the attack of sulphate particles on calcium aluminate hydrate results in the creation of expansive gypsum and ettringite in the concrete matrix. Besides, Monteny et al. [18] described that acidic rain in the urban area is another serious harm that affects the concrete structures. Acid rain is mostly due to the burning of fossil fuels and addition of CO₂ and sulphuric acid to the atmosphere in the form of gases. Therefore, these pollutant gases have harmful influences on concrete components when mixed with rain.

According to Palankar et al. [19], most of the acids can either slowly or rapidly decompose the concrete components, depending on the type and concentration of acid. In the concrete components exposed to acid, Ca(OH)₂ is the most vulnerable constituent. However, other hydration products, such as Calcium-silicate-hydrate (C-S-H) crystals, are also affected by acid. As pointed out by Monteny et al. [18], the deterioration of concrete components exposed to sulphuric acid can be

described through the chemical reactions, as shown in Equations (1)–(3). Consequently, the outcomes of the given reaction between sulphuric acid and concrete are gypsum and ettringite, which are made on the external face of concrete specimens. These harmful products are leads in the creation of cracks and spalling, and therefore, loss in strength of concrete specimens. Prepacked aggregate concrete has a unique construction process, and it has been investigated for its mechanical properties. Nevertheless, there has been a lack of research on the deformation and strength behaviour of prepacked aggregate concrete reinforced with waste carpet fibres and also containing POFA. Given the quarrel as mentioned above, the purpose of this work was to inspect the mutual effects of waste polypropylene fibre and POFA on the strength and deformation properties of new prepacked aggregate concrete, as well as the microstructural analysis. As the construction of PAC requires unique skills and experience that most workers do not have, the current paper offers beneficial information on the manufacturing of PAFRC that can help both engineers and workers. The consumption of waste PP fibre and POFA in the manufacture of PAFRC could be beneficial from both an environmental and economic point of view.

$$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4.2H_2O \tag{1}$$

 $3CaO.Al_2O_{3.12H_2O} + 3(CaSO_{4.2H_2O}) + 14H_2O \rightarrow 3CaO.Al_2O_{3.3}CaSO_{4.32H_2O}$ (2)

$$CaSiO_{2.2}H_{2}O + H_{2}SO_{4} \rightarrow CaSO_{4} + Si(OH)_{4} + H_{2}O$$
(3)

As the development of cracks is one of the major problems of plain concrete exposed to chemical attacks, a substantial technique that declines the brittleness of concrete components is desired. For this purpose, several researchers proposed the reinforcement of concrete with short fibres to enhance the performance of concrete under aggressive environments. Afroughsabet et al. [20] and Mastali and Dalvand [21] specified that by including short fibres into the concrete mix, the brittle nature of concrete enhanced significantly. Besides, Ranjbar et al. [22] and Hossain et al. [23] pointed out that using POFA as cementing materials up to a certain level, moderately improved the acid resistance of concrete by reducing the rate of mass loss and strength loss. Therefore, based on the existing literature, the utilisation of pozzolanic materials and short fibres have been recommended to enhance the performance of concrete exposed to chemical attacks. Amongst all recommended fibres, polymeric based fibres such as polypropylene (PP) are more beneficial in contact with acid and sulphate solutions [24]. Thus, the current paper aimed to investigate the potential use of WMP fibre and POFA in the manufacture of concrete exposed to sulphate and acid attacks. In addition to the compressive strength, the residual properties of concrete exposed to acid and sulphate after one-year exposure were evaluated in terms of visual inspection, mass loss and strength loss. Besides, to assess the effects of chemical attacks on the microstructure of concrete, scanning electron microscopy (SEM) was carried out on the cement passed particles. Moreover, the consumption of waste materials such as plastic wastes in concrete would help to decrease the waste generated, and preserve the environment from pollution.

2. Experimental Program

2.1. Materials

In this study, a type I cement (TASEK CEMENT, Ipoh, Malaysia) that complies with the specifications of ASTM C 150-2007 was used. In addition, OPC was substituted by POFA (PPNJ Kahang Palm Oil Mill, Johor, Malaysia) at a substitution level of 20%. The raw palm oil fuel ash particles were collected as waste from the local mill industry. The ash particles were dried at the temperature of 100 ± 5 °C and then sieved to remove the larger particles of over 150 µm. Subsequently, the small size of ash particles was kept in a crushing machine, and the grinding process was continued for about two hours per each 4 kg of ash. Then, the grounded POFA particles were tested following the specifications of ASTM C618-15 and BS 3892: Part 1-1992 to achieve the desired properties. The ashes which passed the standard requirements with the desired chemical compositions and physical properties, as given in Table 1, were then used as cementing materials.

	Physical		Chemical Composition (%)							
Material	Specific Gravity	Blaine Fineness (cm²/g)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	SO3	LOI *
OPC	3.15	3990	20.3	5.3	4.21	62.5	1.53	0.005	2.13	2.35
POFA	2.42	4930	62.7	4.8	8.14	5.8	3.54	9.07	1.17	6.28

Table 1. Physical properties and chemical composition of ordinary Portland cement (OPC) and palm oil fuel ash (POFA).

* Loss on ignition.

Natural river sand having a specific gravity of 2.6 g/cm³, 2.3 fineness modulus, 0.7% water absorption and a maximum size of 4.75 mm was used. The coarse aggregates of crushed granite with 0.5% water absorption, a specific gravity of 2.7 g/cm³ and a maximum size of 10 mm was used. The polypropylene (PP)-type metalised plastics with aluminium metallisation treatment used in food packing were collected as waste and washed to clean from any contaminations. Subsequently, as shown in Figure 1a–c, the waste films were torn into fibres of 2 mm width and 20 mm length. As the aspect ratio (length/diameter) is an important parameter in the effectiveness of the fibres in the concrete matrix, and due to the rectangular cross-section of WMP fibres, to calculate the aspect ratio, the diameter was determined following the specifications of EN 14889-1:2006 given in Equation (4). Therefore, the fibres with the aspect ratio (l/d) of 47 were used in this study. The typical engineering properties of WMP fibre used in this study are shown in Table 2.

$$d = \sqrt{(4.w.t)/\pi} \tag{4}$$

where w (mm) and t (mm) are the width and thickness of fibre.



Figure 1. (a) Metalised plastic wastes; (b) Preparation of fibres; (c) 20 mm length waste metalised plastic (WMP) fibres.

Table 2. Engineering properties of WMP fibres.

Resin Type	Plastic Type	Size (W * L) (mm)	Density Range (kg/m³)	Thickness (mm)	Tensile Strength (MPa)	Elongation (%)
Polypropylene	LDPE	2 * 20	0.915-0.945	0.07	600	8–10

2.2. Mix Proportions

The mix proportions of different materials used in the production of concrete composites are illustrated in Table 3. In this study, in total, twelve mixtures with different WMP fibre dosages of 0, 0.25%, 0.50%, 0.75%, 1.0% and 1.25% were prepared. Six mixes, namely B1–B6, were made with 100%

OPC, while another six mixes of B7–B12 were cast with 20% POFA as cement replacement. In addition, a water/binder ratio of 0.48 was maintained in all concrete mixes.

Mix	Cement (kg/m ³)	POFA (kg/m ³)	Water (kg/m ³)	Fine Aggregate (kg/m ³)	Coarse Aggregate (kg/m ³)	WMPF (%)
B1	445	-	215	830	860	0.0
B2	445	-	215	830	860	0.25
B3	445	-	215	830	860	0.50
B4	445	-	215	830	860	0.75
B5	445	-	215	830	860	1.0
B6	445	-	215	830	860	1.25
B7	356	89	215	830	860	0.0
B8	356	89	215	830	860	0.25
B9	356	89	215	830	860	0.50
B10	356	89	215	830	860	0.75
B11	356	89	215	830	860	1.0
B12	356	89	215	830	860	1.25

able 5. With details of concrete composite	Table 3.	Mix details	of concrete	composites
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2.3. Production of Specimens

The workability test of the fresh concrete composite was conducted according to BS EN 12350-2:2009 for slump test and BS EN 12390-3:2009 for VeBe time. Besides, cubic concrete specimens measuring 100 mm were fabricated following BS EN 12390-2:2009 and BS EN 12390-3:2009. Following the moulding procedure, the specimens were de-moulded after 24 h and were subsequently placed into a water tank for curing until they were needed for testing purposes. The average ambient temperature recorded in the laboratory was 27 ± 2 °C, where the relative humidity (RH) was $85 \pm 5\%$. Based on the existing literature, there are few techniques to simulate and evaluate the acid and sulphate attacks for plain and fibre reinforced concrete and mortar. Consequently, the immersion method for sulphuric acid and magnesium sulphate in 5% and 10% solutions were adopted in this study, respectively. The immersion procedures were also followed by the methods proposed by Monteny et al. [18] and Sideris et al. [25], who used 10% MgSO₄ solution and 5% H₂SO₄ solution in their studies for these tests.

Similar to the compressive strength test procedure, overall 36 concrete cubes of 100 mm were made for each sulphate and acid tests. After 24 h from casting, the concrete cubes were removed from moulds and kept in water tank for 28 days. Then, the specimens were removed from the water tank and prepared for immersion. The specimens were dried, and the mass in the saturated surface dry (SSD) condition was measured as the initial mass of specimens. The concrete specimens were then submerged in MgSO₄ solution with 10% concentration for the sulphate resistance test, and H₂SO₄ solution of 5% concentration for the sulphuric acid resistance test, for 365 days. The pH values of the solutions were controlled and fixed as 2.5 and 8.5 for sulphuric acid and magnesium sulphate solutions, respectively. Subsequently, the immersed test specimens were removed after 365 days and cleaned by using a dry towel and left in a testing room for about one hour. The visual assessment was done through the critical observation on the shape, size and colour of the specimens immersed in the acid and sulphate solutions and were compared with those cured in water. To evaluate the variation in the mass of specimens, the mass of all concrete specimens were measured and noted as the residual mass after one-year immersion. The percentage of loss in the mass of samples was calculated by using Equation (5):

$$ML_t = \frac{M_t - M_i}{M_i} \times (100) \tag{5}$$

where ML_t is the mass change after 365 days exposure, M_t is the residual mass after the exposure period (gr) and M_i is the original mass before immersion (gr).

In this study, the losses in the strength of concrete specimens after exposure to acid and sulphate solutions were evaluated in terms of strength loss factor (*SLF*) by using Equation (6) as follow:

$$SLF = \frac{F_{cw} - F_{cs}}{F_{cw}} \times (100) \tag{6}$$

where F_{cw} is the cube compressive strength of water-cured companion specimen, and F_{cs} is the cube compressive strength after 365 days exposure in acid and sulphate solutions.

3. Results and Discussion

3.1. Workability

The workability of concrete composites in terms of slump test and VeBe time test was piloted. The outcomes of the workability tests are illustrated in Figure 2a,b. It was discovered that the inclusion of WMP fibres reduced the slump values such that the highest slump value recorded was 190 mm for the control mix (B1), as shown in Figure 3a,b. For the concrete mix reinforced with 0.5% fibre (B3), the slump values recorded were 80 mm. It was clear that the VeBe time increased linearly by adding WMP fibres. Furthermore, the results indicated that the sump of concrete mixes with POFA was less than that of OPC mixes. Likewise, the addition of WMP fibres demonstrated a similar effect on the slump values of POFA mixes. For instance, values of 170 and 50 mm were noted for a slump of B7 and B9 mixtures, respectively. Moreover, the VeBe times increased for the POFA-based concrete, and values of 3.4 and 7.3 s were noted for mixtures of B7 and B9, respectively.



Figure 2. Effects of WMP fibres on (a) slump and (b) VeBe time values.



Figure 3. The slump test of (a) plain OPC mix, and (b) POFA-based mix reinforced with WMP fibres.

3.2. Compressive Strength of Water-Cured Specimens

Figure 4 reveals the values attained from the compressive strength test for concrete mixes at various curing ages. The results showed that the compressive strength dropped by including WMP fibre into the concrete. The outcomes exposed that the obtained values of strength were higher in POFA mixes than those of OPC mixes at the ages beyond 180 days. The reinforcement of OPC concrete mixes by 0.25%, 0.5%, 0.75%, 1% and 1.25% WMP fibres, resulted in the reduction of compressive strength values by about 10.4%, 14.5%, 17.7%, 20.4% and 23.9%, respectively, as compared to the plain control mix (B1) at the age of 28 days. Likewise, at 28 days, in the POFA-based samples with the same fibre content, the compressive strength dropped by 14.6%, 18.8%, 24.7%, 26.4% and 27.9%, respectively. The higher reduction in the strength of POFA mix specimens the early age of 28 days could be due to the slow hydration rate of POFA, which results in lower strength values. Besides, the existence of voids in the concrete specimens, due to the lack of hydration products in the POFA-based specimens as well as the balling effect owing to the higher dosage of fibres, results in comparatively lower strength values [26].



Figure 4. Compressive strength of concrete specimens cured in water.

However, beyond 180 days curing of specimens in water, owing to the consumption of POFA and its high pozzolanic nature, the attained strengths of concrete samples were found to be remarkably greater than those of the early age values. For example, at 365 days, the compressive strength values of 52.9, 51.2, 49.3, 47.6, 44, and 42.5 MPa were recorded for the POFA-based mixes with WMP fibre contents of 0%, 0.25%, 0.5%, 0.75%, 1% and 1.25%, respectively. The values obtained are comparatively

higher than those values of 48.6, 47.2, 44.8, 43.2, 39.8, and 37.4 MPa, which were recorded for the OPC-based mixes with same fibre contents. It could be observed that the POFA mixes show higher strength values, in association with the OPC specimens for the similar fibre contents. The findings of this study are similar to those reported by Ranjbar et al. [22], who described the significant improvement in the compressive strength of concrete by using POFA as a partial cement replacement with longer curing periods.

The microstructural analysis was done using scanning electron microscopy (SEM) to investigate the variations in the hydration process of concrete with the existence of POFA. Figure 5 displays the distribution of C-S-H gel in the pastes at the curing period of 365 days for OPC and POFA mixes. The C-S-H gel was homogeneously dispersed in the paste of POFA mix as related to the OPC paste. The formation of additional C-S-H gels in the POFA mixes due to pozzolnic reactions, as shown in Equations (7) and (8), resulted in a dense microstructure, particularly, and therefore, enhanced the strength of concrete [27]. The reactive silica (SiO₂) present in POFA particles chemically reacted with the liberation of calcium hydroxide as a result of OPC hydration in the presence of moisture, and consequently formed an extra C-S-H gel in the matrix [28]. This reaction was relatively slow; however, the enhancement in the strength of POFA mixes was more evident at the ultimate ages. Besides, Figure 6 displays the interfacial transition zone between fibre and cement paste in the concrete matrix after one year of water curing. The SEM image reveals a robust bond among WMP fibre and blended cement paste. This strong bonding between fibres and paste provides a reliable solid microstructure in the matrix, which results in the reduction in the crack formation at the interface zone. Consequently, a lower volume of cracks were caused in the strength development and, therefore, high ductility and durability performance in aggressive environments was achieved [29].

Hydration reaction of OPC: C₂ or C₃S + H₂O
$$\rightarrow$$
 main C-S-H gel + Ca(OH)₂ (7)

Pozzolanic reaction of POFA:
$$Ca(OH)_2 + SiO_2 (POFA) + H_2O \rightarrow Additional C-S-H gel$$
 (8)



Figure 5. Scanning electron microscopy (SEM) images of OPC- and POFA-based pastes cured in water for 365 days.

3.3. Sulphate Resistance

3.3.1. Visual Inspection

The visual assessment was done on the concrete cubes exposed to 10% MgSO₄ solution for 365 days to assess the effects of sulphate on concrete, as illustrated in Figure 7a,b. In this regard, the changes in the shape, size and the surface colour of the cubical samples were inspected and analysed. The colour of concrete specimens can provide comprehensive information on how the sulphate influenced the

concrete. Table 4 displays the overall visual inspection of the concrete cubes with and without fibres. At the end of the exposure period, a whitish precipitate was detected on the surface of all concrete specimens, which was the result of sulphate attack. However, these precipices were found to be lower on the surface of POFA-based specimens as compared to those of OPC specimens. Due to the higher amount of lime in the OPC specimens, as a result of sulphate attack, the lime particles leached and deposition, and, therefore, calcium sulphate (CaSO₄) was formed in higher quality, and the whitish precipitate was formed on the outer surface of specimens [30]. It was also observed that cracks were formed on the surface of plain concrete specimens.



Figure 6. Interfacial transition zone with strong bonding between paste and fibre cured in water for 365 days.



Figure 7. Concrete specimens of (**a**) OPC-based and (**b**) POFA-based mixes at 365-day of exposure in MgSO₄ solution.

Mix	Surface Texture	Size	Colour	Edge	Shape
OPC					
B1	Slightly deteriorated	Marginally increased	Whitish deposit	Fine cracks	Perfect cube
B2	Smooth	Marginally increased	Whitish deposit	Fine cracks	Perfect cube
B3	Smooth	No change	Whitish deposit	Perfect	Perfect cube
POFA					
B7	Slightly deteriorated	Marginally increased	Light grey	Fine cracks	Perfect cube
B8	Slightly deteriorated	Marginally increased	Light grey	Fine cracks	Perfect cube
B9	Smooth	No change	Light grey	Perfect	Perfect cube

Table 4. The outcomes of visual inspection of typical cubical concrete samples exposed to $MgSO_4$ solution for 365 days.

Although the cracks detected on the POFA specimens were found to be finer than those cracks developed on the OPC specimens, in the reinforced specimens with 0.25% and 0.5% fibres very fine cracks were formed in both OPC and POFA mixes. From the observations made, it can be concluded that the inclusion of WMP fibres caused in the superior prevention of crack development on the surface of the specimens. This could be owing to the interlocking of concrete constituents with the help of fibres, which arrest the microcracks and prevent the further formation of cracks [31]. It was also noted that no physical distortion was found on the specimens reinforced with WMP fibres, and the specimens with POFA performed better due to the more extended period of test and excellent pozzolanic activity of POFA after one year.

3.3.2. Mass Gain

In this study, the influence of sulphate attack on the variation in the mass of concrete specimens after one year of immersion was assessed. Figure 8 displays the changes in the mass of cubical samples at one year of immersion in a sulphate solution. A similar tendency was observed for all specimens reinforced with WMP fibres, in which the masses of all specimens were gained by absorbing the sulphate particles after one year of exposure. It can be seen that the masses of plain OPC and POFA concrete mixes gained by about 1.56% and 0.9%, respectively. Nevertheless, the addition of WMP fibres up to 1% fibre content into the concrete mix considerably prevented the gain in the mass of the cube samples. For instance, the masses of OPC-based concrete specimens reinforced with 0.25%, 0.5%, 0.75% and 1% fibres gained by 0.53%, 0.6%, 1.1% and 1.43%, respectively, whereas for the POFA-based specimens the masses gained by 0.38%, 0.545, 0.88% and 1.2% for the same fibre content, respectively. The outcomes exposed that the substitution of OPC by 20% POFA resulted in a significant reduction in the mass gain of concrete specimens with and without fibres. However, a further increase in the fibre content beyond 1% increased the mass gain of the samples immersed in a sulphate solution. The lower percentage of mass gain in the specimens reinforced with WMP fibres could be due to the development of a grid structure in the matrix by fibres, which prevented the diffusion and disruption of harmful constituents into the concrete specimens. Therefore, using POFA as a partial cement replacement in concrete under a sulphate environment is beneficial to provide a dense microstructure and filled up the pores in the matrix by superior pozzolanic action [32,33].



Figure 8. Effect of WMP fibre on the mass gain of concrete specimens exposed to MgSO₄ solution.

3.3.3. Residual Compressive Strength

In this study, to assess the effects of the sulphate attack on the strength performance of concrete, the residual compressive strength of specimens after one-year immersion in MgSO₄ solution was carried out. The results of the test and comparison amongst the water-cured specimens and those specimens exposed to sulphate solutions are demonstrated in Figure 9. Besides, the variation in the strength values of specimens cured in water and those exposed to sulphate attack was termed strength loss. It was noted that the compressive strength of all concrete mixes was affected by sulphate attack. When the companion samples gained their strength, and the hydration process was continued in the water at room temperature, the test samples were suffered in sulphate solution and lost their strength. The results obtained also indicated that the rate of strength losses was higher in OPC-based concrete specimens than those of POFA-based specimens for all fibre volume fraction mixes. For instance, the strength loss values of 10.6, 7.1, 5.6, 10.8, 13.8 and 16.3 MPa were recorded for OPC-based specimens reinforced with 0%, 0.25%, 0.5%, 0.75%, 1% and 1.25% fibres, respectively, while in POFA-based specimens, comparatively lower strength loss values of 8.6, 6.3, 4.1, 9.4, 11.8, and 13.5 MPa were noted for the same fibre dosages, respectively. This reduction in the strength of all mixes was due to the long-term immersion of specimens in the MgSO₄ solution. Moreover, the formation of cracks in the OPC-based specimen as a result of sulphate attack leads to accelerate the loss in strength of concrete. However, the combination of POFA and WMP fibres minimised the development of cracks and prevented the spalling of concrete specimens. The finding of this study is comparable to those results reported by Al-Rousan et al. [34] on the potential use of polypropylene fibres against the sulphate attack in concrete.

The strength loss factor (SLF) of concrete mixes owing to the deterioration by sulphate attack was also evaluated. The outcomes of SLF after one year of exposure to the sulphate solution are demonstrated in Figure 10. It was observed that SLF developed for all concrete mixes, which indicates the effect of sulphate attack on the strength of concrete. The results show that the SLF values are comparatively higher for the OPC-based specimens as compared to those of POFA mixes. It is interesting to note that a lower SLF value was found for the POFA mix containing 0.5% WMP fibre, at 8.2%. However, the SLF values increased with the rise in fibre content.



Figure 9. Effect of WMP fibres on the compressive strength of water-cured specimens and those exposed to MgSO₄ solution after 365 days.



Figure 10. Strength loss factors of concrete mixes exposed to MgSO₄ solution at 365 days.

To assess the influence of MgSO₄ solution on the microstructure of concrete specimen, the SEM test was carried out. As revealed in Figure 11, the nature of paste specimens has changed due to the sulphate attach as compared to that SEM image of the water-cured specimen at the age of 365 days (Figure 5). The SEM images showed that the voids in the OPC paste were slowly occupied with newly precipitated flakes as a consequence of MgSO₄ contact. Therefore, a weak microstructure in the concrete matrix was formed, which caused the reduction of strength values as well as the low

durability performance [34,35]. However, with the presence of POFA in the concrete mixture, due to the superior pozzolanic action, mostly with longer curing periods, additional hydration products such as C-S-H gel were formed. These C-S-H gels then filled up the existing cavities in the matrix, and, consequently, there was no place for the new harmful products, resultant from the sulphate attack. As the C_3A content is higher in the OPC specimens, theoretically, the matrix is more prone to attacks by sulphate solution. However, with the existence of POFA, and due to the pozzolanic reactions given in Equations (6) and (7), the Ca(OH)₂ in OPC chemically reacted with active SiO₂ present in POFA and formed additional C-S-H gels, which prevent Ca(OH)₂ reacting with MgSO₄ solution to create gypsum and ettringite. Therefore, with a lower amount of gypsum and ettringite, the performance of concrete enhanced and improved the resistance against sulphate attacks [36].



Figure 11. SEM of OPC and POFA paste exposed to sulphate solution for 365 days.

3.4. Acid Resistance

3.4.1. Visual Inspection

In this study, to assess the effects of H_2SO_4 solution on the concrete specimens, a visual inspection was performed. Generally, a visual observation which includes changes in colour, spalling, crack formation on the surface of specimens and deposit of new precipices on the surface is the first step to evaluate the effects of the acid solution on the concrete. Figure 12a,b illustrates the concrete specimens exposed to H_2SO_4 solution for one year. As shown in Figure 13, the colour of concrete offers comprehensive information on the influence of acid on concrete matrix; whether the colour implies the initially contacted surface or one resultant acid attack. It was detected that the shape and surface texture of plain specimens without fibres deteriorated as a result of the acid attack. However, slight changes in the shape and colour of specimens reinforced with WMP fibres were detected. The observation made indicates that the size and shape of the POFA-based specimens with WMP fibres remained same as the original due to the high resistance of POFA particles against acid attack, which prevented spalling and washed out the specimens, while the size of the OPC-based specimens were reduced by spalling and washing out as a result of acid attack. The visual assessment also revealed that with the addition and increase in the fibre dosage, the resistance against acid increased significantly. This superior resistance against acid attack was more evident in the POFA-based specimens. In general, lower spalling and changes in the shape were observed in the concrete mixes containing WMP fibres compared with those of plain mixes. This might be owing to the linking action of WMP fibres, which prevented the washing out and spalling of concrete attacked by acid, and thus, enhanced the performance of concrete in aggressive environments [37,38].

Figure 12. Concrete specimens of (**a**) OPC-based and (**b**) POFA-based mixes exposed to H₂SO₄ solution for 365 days.



Figure 13. Cross-section of concrete specimen exposed to H₂SO₄ solution.

3.4.2. Mass Loss

The changes in the mass of concrete cubes submerged in an acid solution for one year are shown in Figure 14. A similar trend of mass loss was observed for all mixes after one year of exposure to H_2SO_4 solution. The results show that the rate of mass losses in OPC-based specimens was comparatively higher than those of POFA-based specimens. It indicates that the OPC specimens washed out at a higher rate when immersed in an acid solution. This higher rate of leaching in the OPC specimens might be due to the existence of CaO at a higher volume in the OPC than POFA, which quickly washed out when coming into contact with acid [39,40]. However, by reinforcing OPC specimens with WMP fibres, the rate of mass loss decreased significantly. For instance, by adding fibres of 0.25%, 0.5%, 0.75%, 1% and 1.25%, the mass losses of 14.1%, 10.5%, 8.4%, 7.1% and 5.9% were recorded, respectively, which are comparatively lower than that of 19.6% mass loss noted for plain OPC mix without any fibre. Moreover, the rate of mass losses in the POFA mixes containing WMP fibres was considerably

lower than those of OPC reinforced mixes. The mass losses of 11.3%, 8.7%, 6.4%, 4.2% and 3.7% were recorded for the fibre dosages of 0.25%, 0.5%, 0.75%, 1% and 1.25%, respectively, which are all lower than that of 16.8% recorded for plain POFA-based mix. With the addition of WMP fibres, through the bridging action of fibres the aggregates and cement past connected with a strong bond, and therefore, degradation and spalling of concrete specimens significantly reduced.



Figure 14. Effect of WMP fibres on the mass loss of concrete specimens exposed to the acid solution for 365 days.

3.4.3. Residual Strength

The outcomes of the residual compressive strength after 365 days of immersion in H₂SO₄ solution are shown in Figure 15 and compared with those mixes cured in water. Due to the long-term exposure to the acid solution, strength loss was revealed in all mixes. The results revealed that the losses in compressive strength loss, which is the difference between the strength of the water-cured specimens and those exposed to the acid solution, decreased with addition and a further increase in the WMP fibres. Moreover, the strength losses were observed to be higher for the OPC-based specimens than those of the POFA mixes. For the OPC mixes, the addition of WMP fibre at the dosages of 0.25%, 0.5%, 0.75%, 1% and 1.25% caused the strength losses of 28.9, 26.1, 24.5, 22.8 and 21.9 MPa, respectively. It can be seen that all of the recorded values were lower than that of 33 MPa obtained for plain concrete mix, which indicates the positive effect of fibres in controlling the strength loss. Besides, comparatively lower strength losses of 26.7, 24, 22.7, 21.5 and 20.9 MPa were noted for POFA mixes with the same fibre dosages, which are all lower than that of 29.8 MPa recorded for plain POFA mix. As revealed in Figure 15, those mixes with both WMP fibres and POFA obtained lower strength losses. This improvement was predictable due to the existence of POFA as a partial cement replacement, which contains a lower CaO content compared to OPC. The lower amount of CaO, which quickly dissolve in acid, results in the reduction of washing out and spalling of concrete specimens [41–43]. Besides, the existence of WMP fibres prevented the development of cracks by bridging action and reduced the spalling of concrete samples. Therefore, the mutual effects of POFA and WMP fibre lead to enhancements in the strength and durability performance of concrete mixtures exposed to acidic environments.



Figure 15. Effect of WMP fibres on the compressive strength of water-cured specimens and those exposed to H₂SO₄ solution after 365 days.

The SLF values of concrete mixes after one year of contact with acid were also evaluated, and the outcomes are shown in Figure 16. It can be perceived that the SLF values of POFA mixes were relatively lower than those values recorded for OPC mixes. As mentioned earlier, the better performance of POFA-based specimens in the acid environment could be due to the superior pozzolanic nature of POFA, which produced additional hydration products of C-S-H gel by consumption of Ca(OH)₂. These additional gels then filled up the porosities in the matrix and prevented the entrance of harmful particles into the matrix and, therefore, enhanced the performance of concrete [23].



Figure 16. The strength loss factor of concrete mixes after 365-day immersion in the acid solution.

The microstructure of concrete mixes exposed H_2SO_4 solution for the one-year period are demonstrated in Figure 17. As revealed in the SEM images, the gypsum (Ca₂SO₄.2H₂O) was formed in large quantities during the exposure period. However, the gypsum particles were found to be lower in the POFA mix compared to that of OPC mix. Generally, soft and soluble gypsum is one of the main products of the chemical reactions between cement particles and sulphuric acid [42,43]. Owing to the lesser quantity of Ca(OH)₂ in POFA mixes which chemically reacts in the pozzolanic reaction, the formation of gypsum reduced. Consequently, the crack development and spalling reduced and results in the more durable and denser microstructure of POFA specimens against acid attack.



Figure 17. SEM images of OPC and POFA paste exposed to the acid solution for 365 days.

4. Conclusions

Considering the growth in the green production by using solid wastes in the production of concrete, which is vulnerable in the aggressive environment, the current study critically investigated the performance of concrete containing POFA and WMP fibres exposed to sulphuric acid and magnesium sulphate solutions. The following are the conclusions made based on a visual inspection and experimental results:

- (1) The inclusion of WMP fibre reduced the workability of all concrete mixes, with lower slump values and higher VeBe times compared with those of the plain mix.
- (2) The cube compressive strength of water-cured specimens was reduced by adding WMP fibres. However, the compressive strength significantly developed with the presence of POFA after 365 days.
- (3) In sulphate solution, enhanced performance in the resistance of concrete mixes to sulphate attacks was attained by utilising WMP fibres and POFA. The main consequence of fibres was attributed to the formation of the grid structure to avoid the entering of destructive particles into the matrix and crack formation. Besides, the lower rate of mass gain and strength losses against sulphate attacks were detected for reinforced mixes.
- (4) A satisfactory level of resistance to the sulphuric acid attacks was observed for the fibre reinforced specimens. The primary effect of POFA was attributed to the dilution effect of pastes owing to the replacement of OPC by POFA, which decreased the existing amount of Ca(OH)₂ content for reaction with acid and formation of gypsum. Besides, lower mass and strength losses were noted for the mixes containing POFA and WMP fibres.
- (5) The microstructural analysis of paste specimens revealed changes in the nature of hydration products as a result of acid and sulphate attacks. However, the POFA-based specimens performed better against chemical attacks compared with those of OPC mixes owing to the development of supplementary C-S-H gels as a result of the pozzolanic reaction, which filled up the voids in the

matrix and provided a denser microstructure. It, therefore, enhanced the performance of concrete in terms of strength and durability under chemical attacks.

(6) The utilisation of plastic waste fibres and POFA has potential in retaining the integrity of the concrete matrix under chemical attacks. Additionally, conservation of waste materials and lower environmental contamination would also be achieved.

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