Advances in Materials Research, *Vol. 12, No. 3 (2023) 227-242*
https://doi.org/10.12989/amr.2023.12.3.227

Experimental and numerical study on the mechanical properties of reinforced polyester composites

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(Received October 6, 2021, Revised April 27, 2023, Accepted April 28, 2023)

Abstract. Polyester composites play a vital role in civil engineering applications, especially in bridge and car park structures. Therefore, the addition of waste silica-based fillers will both improve the mechanical and durability performance of composites and produce an environmentally friendly material. In this study, the mechanical performance of polyester composites was investigated experimentally and numerically by adding micro and nano-sized silica-based fillers, marble powder, silica fume and nano-silica. 24 cubes for the compression test and 18 prisms for the flexural test were produced in six different groups containing 30% marble powder, 5% silica fume and 1% nano-silica by weight. SEM/EDS testing was used to investigate the distribution of filler particles in the matrix. Experimentally collected results were used to validate tests in the Abaqus software. Additionally, the Extended Finite Element Method (XFEM) was used to estimate the fracture process for the flexural test. The results show that the added silica fume, marble powder and nano silica improves the compressive strength of polyester composites by 32-38% and the flexural tensile strength by 10-60% compared to pure polyester composite. The numerically obtained results matched well with the experimental data, demonstrating the accuracy and feasibility of the calibrated finite element model.

Keywords: crack propagation; FEM; mechanical properties; polyester composites; SEM/EDX

1. Introduction

The use of polyester composites in engineering projects has increased due to their superiority over cement-based construction materials (e.g., high toughness, fast curing, high strength, and tightness) (Elalaoui *et al.* 2018, Ahmed and Khanna 2020, Akaluzia *et al.* 2020). The reinforcement can be waste materials, fibers or nano additives (Lokuge and Aravinthan 2013, Daghash *et al.* 2016, Elalaoui *et al.* 2018, Dębska *et al.* 2020). A large amount of waste is generated every year, which causes air and groundwater pollution. For this reason, the disposal of industrial wastes requires a significant cost.

There have been concerted efforts to beneficially use waste materials in civil engineering applications. The use of such material, as a partial substitute of cement or polymer resin, will

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http://www.techno-press.org/?journal=amr&subpage=5 ISSN: 2234-0912 (Print), 2234-179X (Online)

culminate in strength, durability economic and environmental benefits for the construction industry.

In polyester composites, the filler acts as reinforcement and the polyester acts as the matrix phase. Generally, the stiffness of the reinforcement phase is greater than that of the matrix phase; when combined together produces better mechanical properties.

Researches have been done in the past for the use of some industrial by-products, such as marble powder, silica fume, etc., in concrete (Bostanci 2020, Das *et al.* 2020, Ince *et al.* 2020, Shukla *et al.* 2020, Boukhelf *et al.* 2021, Chand *et al.* 2021, Choudhary *et al.* 2021, Danish *et al.* 2021, Liu *et al.* 2021, Nasier 2021, Zhang *et al.* 2021).

In addition to these, commercial wastes such as red mud, fly ash, blast furnace slag and, etc., have been used in conjunction with polymer composites (Pradeep 2015, Wawrzeńczyk *et al.* 2016, Mo *et al.* 2017, Kucukdogan *et al.* 2018, Vandevenne *et al.* 2018, Gameiro *et al.* 2020, Chaturvedi *et al.* 2021, El Alouani *et al.* 2021). Recently, one of the waste materials preferred to improve the properties of polymer composites is silica fume and marble powder due to their environmental and economic advantages (Benzannache *et al.* 2018, Rokbi *et al.* 2019, Awad *et al.* 2020).

Awad and Abdellatif (2019), found that the thermal and mechanical properties of the low density polyethylene matrix were improved when marble dust particles were added. Çınar and Kar (2018), concluded that composites obtained from PET/Marble powder showed better mechanical, thermal and morphological properties than sole waste PET material. Awad *et al.* (2019), concluded that the addition of marble dust particles improves the thermal and mechanical properties of polypropylene matrix composites. Mansour *et al.* (2017), the flexural test results of polymer concrete reinforced with natural alpha fibers and marble dust showed that concrete containing 20% marble was stronger and more rigid.

Similarly, silica fume fillers favorably improve the mechanical properties of polymer-based matrices, as demonstrated by (Raja and Kumaravel 2015) that the mechanical properties of the Nylon 66 matrix are improved by adding silica fume.

The previous literature review pointed to the positive contribution of silica-based materials to the mechanical properties of polymer-based composites, and also gave us the chance to produce sustainable eco-friendly materials.

Therefore, in this study, it is aimed to fill the gap in the literature and develop a polyester composite with high performance properties by using micro and nano-sized silica-based fillers, marble dust, silica fume and nano-silica. Thus, the effect of both the content and size of the filler will be examined. Powders were used individually or in combination to examine the effect of dual uses on the mechanical properties of polyester composites. Experimentally collected results were used to validate the tests in the Abaqus software, and the Extended Finite Element Method (XFEM) was used to estimate the fracture process for the flexural test.

2. Materials and method:

2.1 Materials

In this work, waste marble powder and silica fume were obtained from the province of Afyonkarahisar in Turkey (Fig. 1) and the index and chemical properties are summarized in Table 1. Whereas, Table 2 summarizes the properties of the n-SiO_2 used.

Table 1 Chemical and index properties of silica fume and marble powder

Chemical composition (%)	Silica fume	Marble powder
SiO_2	88.8	14.25
Al_2O_3	0.17	-
Fe_2O_3	0.11	4.90
CaO	1.01	-
MgO	0.88	-
SO_3	0.42	-
Na_2O	0.19	-
K_2O	5.08	-
$CaCo_3$	-	57.4
Index properites		
Specific gravity	2.36	2.70
Specific surface (cm^2/g)	302	-

Table 2 Properties of n-SiO_2

Purity (%)	+99.5			
Average particle size (nm)	15-35			
Specific surface area (m^2/g)	150-550			
Bulk density (g/cm^3)	<0.1			
True density (g/cm^3)	2,2			
Elemental analysis (%)	Fe	C_a	T_i	N_a
	0.002	0.007	0.012	0.003

Fig. 1 Materials used in the preparation of composite samples

2.2 Fabrication of composites

In this study, unsaturated polyester resin was used in the preparation of polyester composite samples. 10 g of methyl ethyl ketone peroxide (MEKP) and 2 g of cobalt were used per 1 kg of polyester to ensure full hardening of the polyester matrix and to allow faster decomposition and reaction of the hardener. As seen in Table 3, micro and nano-sized silica-based particles, marble dust, silica fume and nano-SiO_2 were added as polyester filler reinforcement. For each group, four $50 \times 50 \times 50$ mm cube specimens and three $40 \times 40 \times 160$ mm prism specimens were cast for compression and flexural tests, respectively. Fig. 2 shows the sample mixing procedure.

Table 3 Mixing ratios of polyester composites

Group	Polyester, %	Marble powder, %	Silica fume, %	Nano-silica, %
PR	100	-	-	-
PS	95	-	5	-
PM	70	30	-	-
PN	99	-	-	1
PSN	94	-	5	1
PMN	69	30	-	1

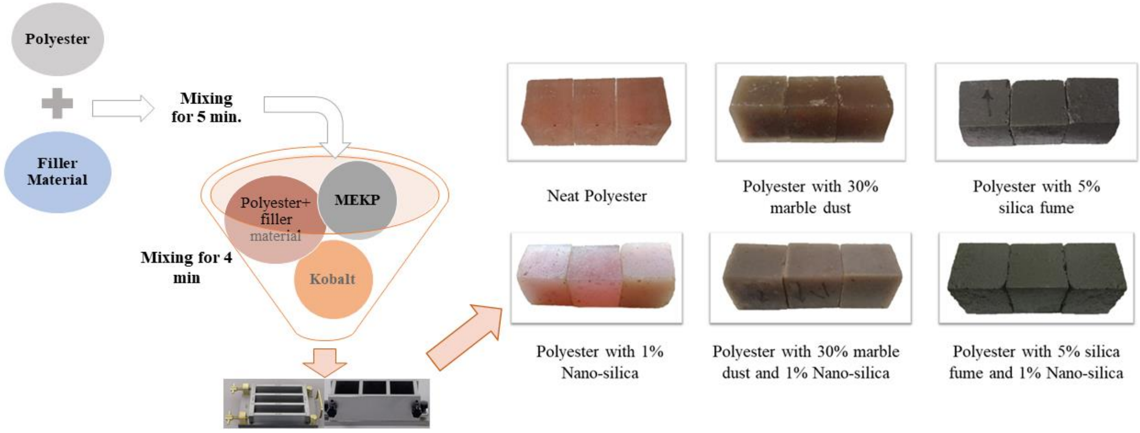


Fig. 2 Sample mixing procedure

2.3 Compressive and flexural tensile tests

A 3000-ton capacity compression tester was used to perform the compression and flexural tests. Compression and flexural tests were performed according to TS EN 12390-3 and TS EN 12390-5 standards, respectively. A constant pressure of 0.4 MPa/s for compression and 0.04 MPa/s for flexural was applied during the test.

2.4 Scanning Electron Microscopy (SEM/EDX)

SEM/EDX technique was used to examine the microstructure of polyester composites. A Quanta FEG 250 scanning electron microscope was used for this purpose. SEM images were taken with magnification factors of 8000x and 10000x.

2.5 Numerical model

Fig. 3 shows the meshing, loading and boundary conditions for compression, fatigue and flexural tests. In the compression test, cube specimens with 50 mm edge width were modeled using the C3D8R element. A 2 mm thick rigid plate was placed over the samples to ensure uniform distribution of the applied displacement. The lower surface of the finite element model was fixed support (i.e., U1=U2=U3=UR1=UR2=UR3=0) and the upper surface was displacement rate controlled.

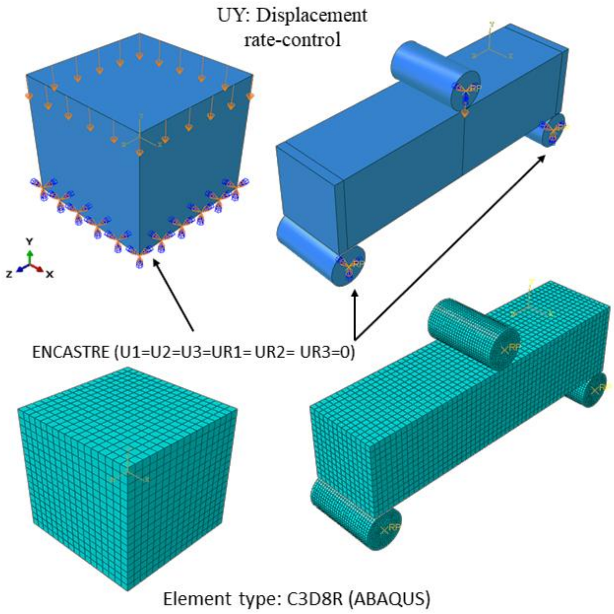


Fig. 3 FE model and its meshing, loading and boundary conditions

In the flexural test, a $40 \times 40 \times 160$ mm rectangular prism specimens were modeled using XFEM to simulate the fracture process. A displacement rate-controlled loading was applied in the middle of the span. Both compression and flexural tests were performed under static loading.

2.6 Identification of material parameters

The complicated and complex simulations demand proper input to build and verify finite element models with experiments (Petrík and Ároch 2019). The true stress-strain concept is important for dealing with large plastic deformation of materials. The engineering stress-strain curve obtained from the experimental tests was converted into a true stress-strain curve using logarithmic equations. This procedure can be utilized in elastic zone and partially in plastic zone from the yield point to ultimate strength point. The total true strain produced during the tensile test was calculated as in Eq. (1).

$$\varepsilon = \ln\left(\frac{l}{l_0}\right) = \ln(1+e) \quad (1)$$

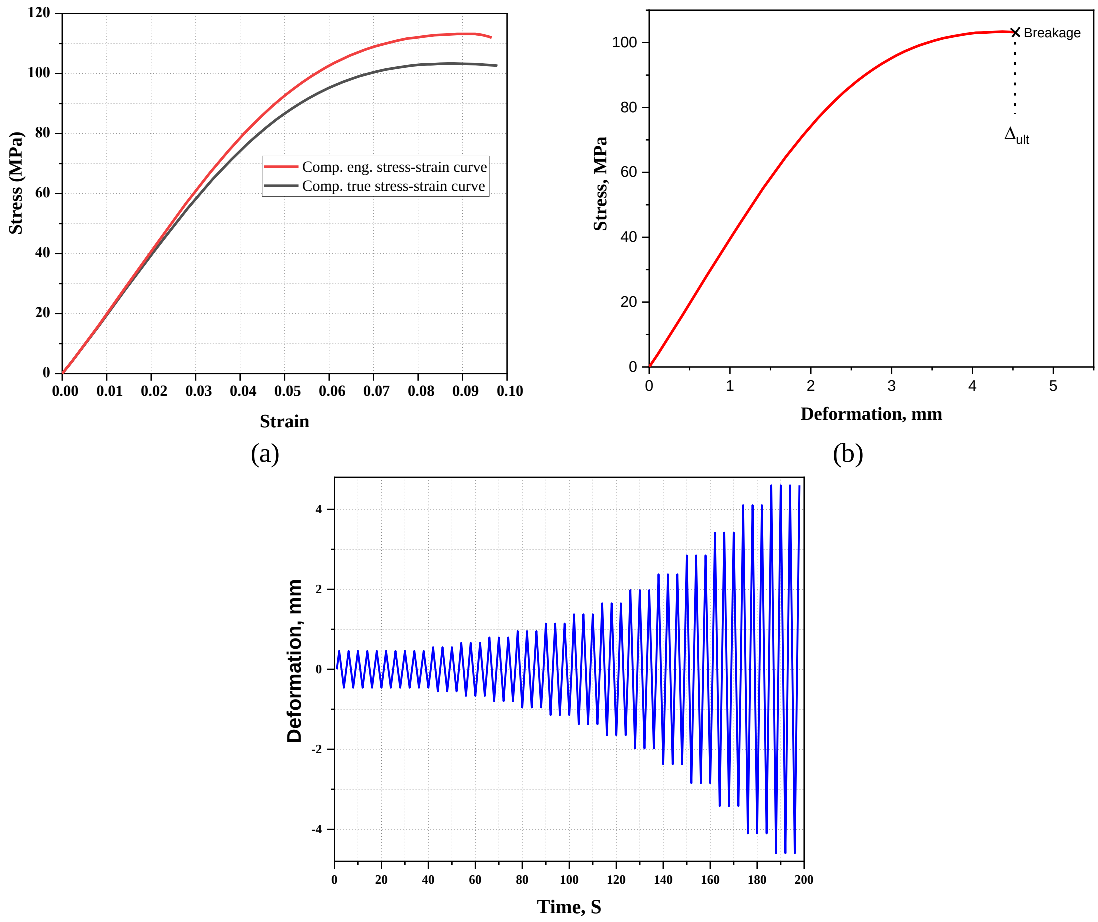
Where, l and l_0 are the current and initial lengths of a tensile sample and, e is the engineering strain

Similarly, compression case, the engineering strain is of magnitude $e = (h_0 - h)/h_0$, while the magnitude of the true strain is

$$\varepsilon = \ln\left(\frac{h_0}{h}\right) = -\ln(1-e) \quad (2)$$

Where, h_0 and h are the initial and current height of specimen,

In the case of compression, the true stress becomes lower than the engineering stress, and in the case of tension, it becomes higher than the engineering stress; however, the true strain is higher



(c)

Fig. 4 (a) Typical monotonic stress-strain curve of polymer composites (b) Monotonic Stress-deformation test for neat polyester composite (c) Loading protocol according to FEMA 461

than the engineering strain in compression and smaller than the engineering strain in tension

The correlation between the engineering stress (s) and the true stress (σ) is found in Eq. (3)

$$s = \sigma \exp\left(\mp\varepsilon\right) \tag{3}$$

Here, the positive sign corresponds to compression and the negative sign to tension.

In this study, polyester composites were defined as elastoplastic materials. In this particular, the compression true stress-strain curves were calculated (Yang *et al.* 2013, Karimi *et al.* 2017, Chen *et al.* 2020). Fig. 4(a) shows the engineering and true stress-strain curve of the reference polyester composite group. The true stress-strain curves for all polyester composite groups were calculated as described above and defined in the ABAQUS software.

2.7 Loading protocol

The loading protocol recommended by FEMA 461 was used in this study. The following steps

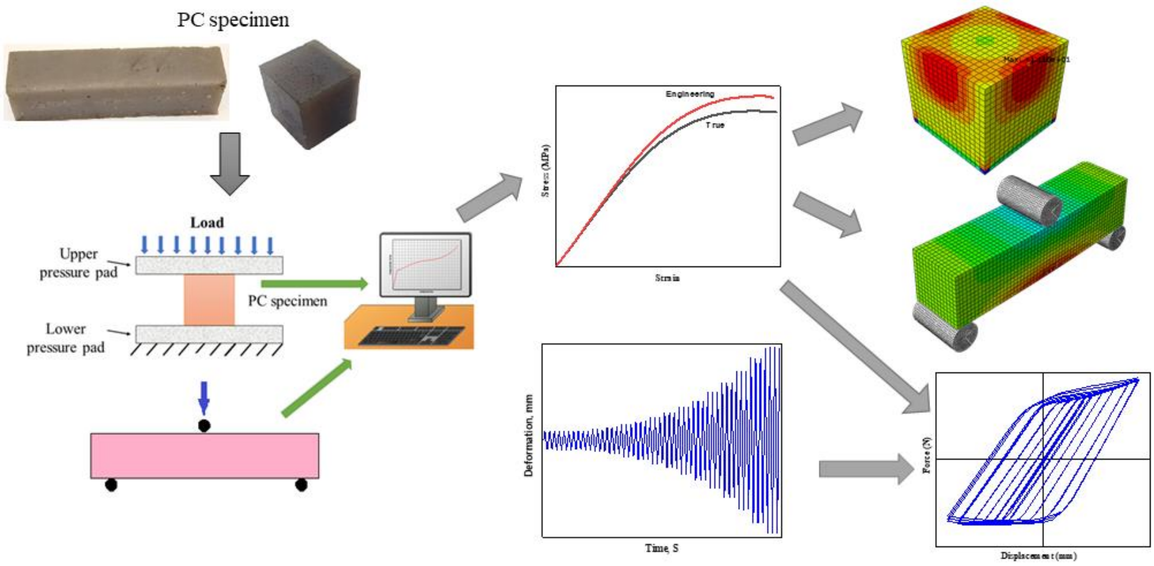


Fig. 5 Experimental and numerical test procedure

were used to obtain the load protocol curve for polyester composites:

- The monotonic stress-deformation test on the polymer composites was conducted (Fig. 4b). Then the deformation at which the samples undergo complete damage (Δ_{ult}) were estimated. The cyclic test was started at 1/10th of the ultimate deformation, $\Delta_1 = \Delta_{ult}/10$. In the first step of the cyclic test, 10 cycles of deformation amplitude Δ_1 were applied.
- For the 2nd stage of the cyclic test, the deformation amplitude increased by 20%, i.e., $\Delta_2 = 1.2\Delta_1$. The samples subjected to three cycles of deformation amplitude, Δ_2.

For each subsequent step, the deformation amplitude increases by 20% and subjected the sample to three deformation cycles. The cyclic testing continued in this manner until the component was completely damaged. Fig. 4(c) shows loading protocol calculated. The experimental and numerical procedure for this article is summarized in Fig. 5.

3. Results and discussion

3.1 Compressive strength

A 28-day compressive strength test was performed for four cubes in each group. The results of the average compressive strength value of pure/reinforced polyester composite samples are shown in Fig. 6. When individual and combined silica fume, marble powder and nano-silica were added, the compressive strength of polyester composites increased by 32-38%. The highest compressive strength was noticed in the PSN and PN groups. Nano silica when added alone and combined with silica fume has a positive effect on compressive strength, while the combination of nano silica with marble powder slightly reduces the compressive strength.

In Fig. 9(a)-(f), the stress-strain curves show that the addition of marble powder increases the ductility of polyester composites, while nano-silica and silica fumes produce brittle samples.

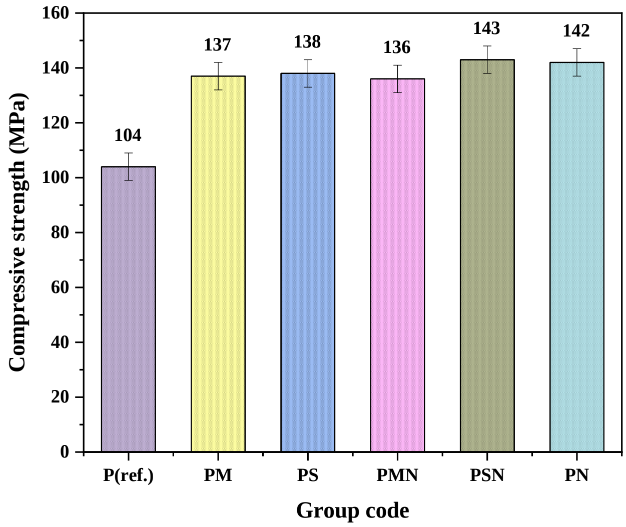
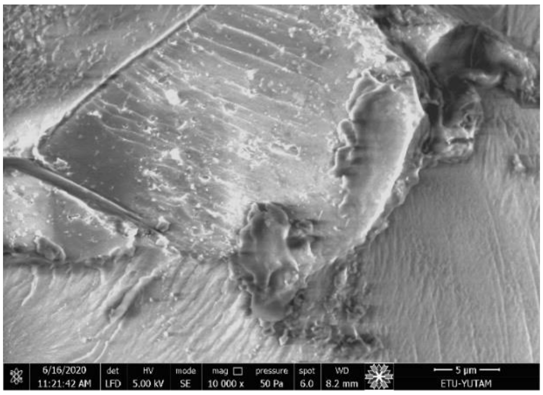
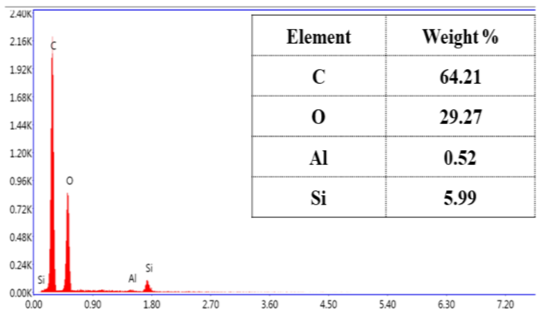


Fig. 6 Results of experimental compressive strength

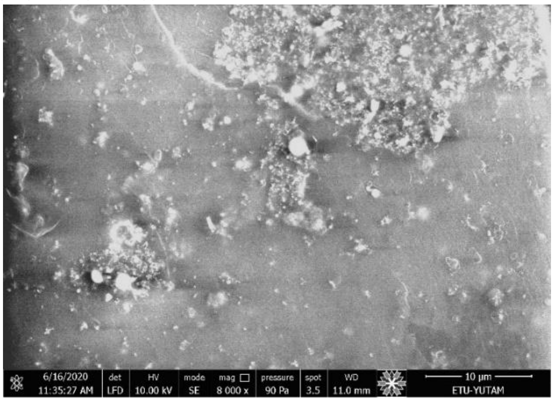


(a) SEM images for specimen with marble powder and nano-silica

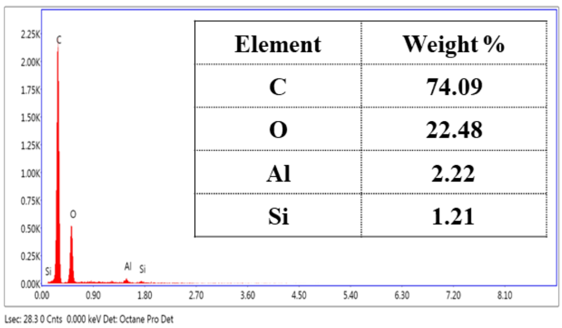


Element	Weight %
C	64.21
O	29.27
Al	0.52
Si	5.99

(b) EDX for specimen with marble powder and nano-silica



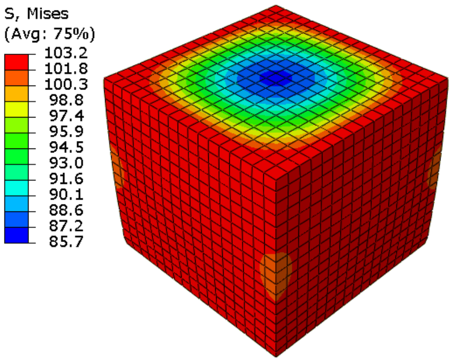
(c) SEM images for specimen with silica fume and nano-silica



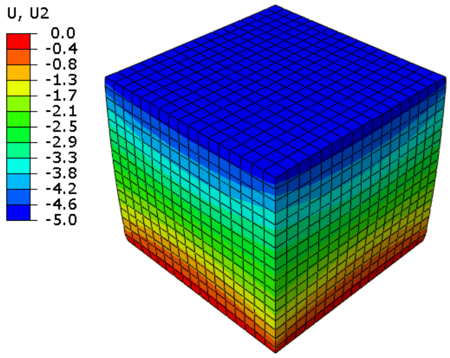
Element	Weight %
C	74.09
O	22.48
Al	2.22
Si	1.21

(d) EDX for specimen with silica fume and nano-silica

Fig. 7 SEM and EDX analysis



(a) Mises stress distribution for PR



(b) displacement in the loading direction for PR

Fig. 8 Numerical compressive test analysis results for reference sample

Scanning electron microscopy (SEM) was used to investigate the distribution of filler particles in the matrix. SEM images show that these particles increase the roughness of the fractured surface, thereby increasing the strength (Figs. 7(a) and (c)). Since nanoparticles have superior properties due to their large surface area, the addition of nanomaterials was expected to increase the compressive strength to a value higher than the value found in this study, but as shown in Figure 7c, agglomeration occurred during sample preparation and therefore weak zones were formed.

EDX spectrums Fig. 7(b) and (d) show that samples containing marble dust, silica fume, and nano-silica have high carbon and oxygen content and small amounts of aluminum oxide (Al_2O_3) and silicon dioxide (SiO_2).

The mechanical behavior of neat/reinforced polyester composites was investigated in FE models. The stress-strain curves calculated in the FE model were compared with the experimental results. Fig. 9(a)-(f) shows that the numerically calculated results match well with the experimental results. Mises stresses and displacement of the neat polyester composite in the loading direction are shown in Figs. 8(a) and (b). Beginning from the calibrated and validated model, it is currently conceivable to perform different tests on similar polyester samples, or on different samples utilizing a similar polyester; however, various dimensions or shapes, with a low financial cost and time-consuming.

3.2 Flexural tensile strength

Fig. 10(a) clearly shows that the addition of individual or combined silica fume, marble powder, and nano-silica significantly improved the flexural tensile strength of polyester composites. The adhesion between the matrix and the reinforcement particles plays a vital role in load transfer. In other words, poor adhesion causes loading transfer to fail. SEM images (Fig. 7) show that the reinforced samples have rough fracture surface and good adhesion with the polyester matrix. Therefore, during the crack propagation process, the crack tip matches with the filler particles and changes its path. This can prevent crack propagation and the fracture energy is increases during the damage process.

In 1999 (Belytschko and Black 1999) developed the extended finite element method (XFEM). This method, which is based on the principle of the division of unity and allows the existence of

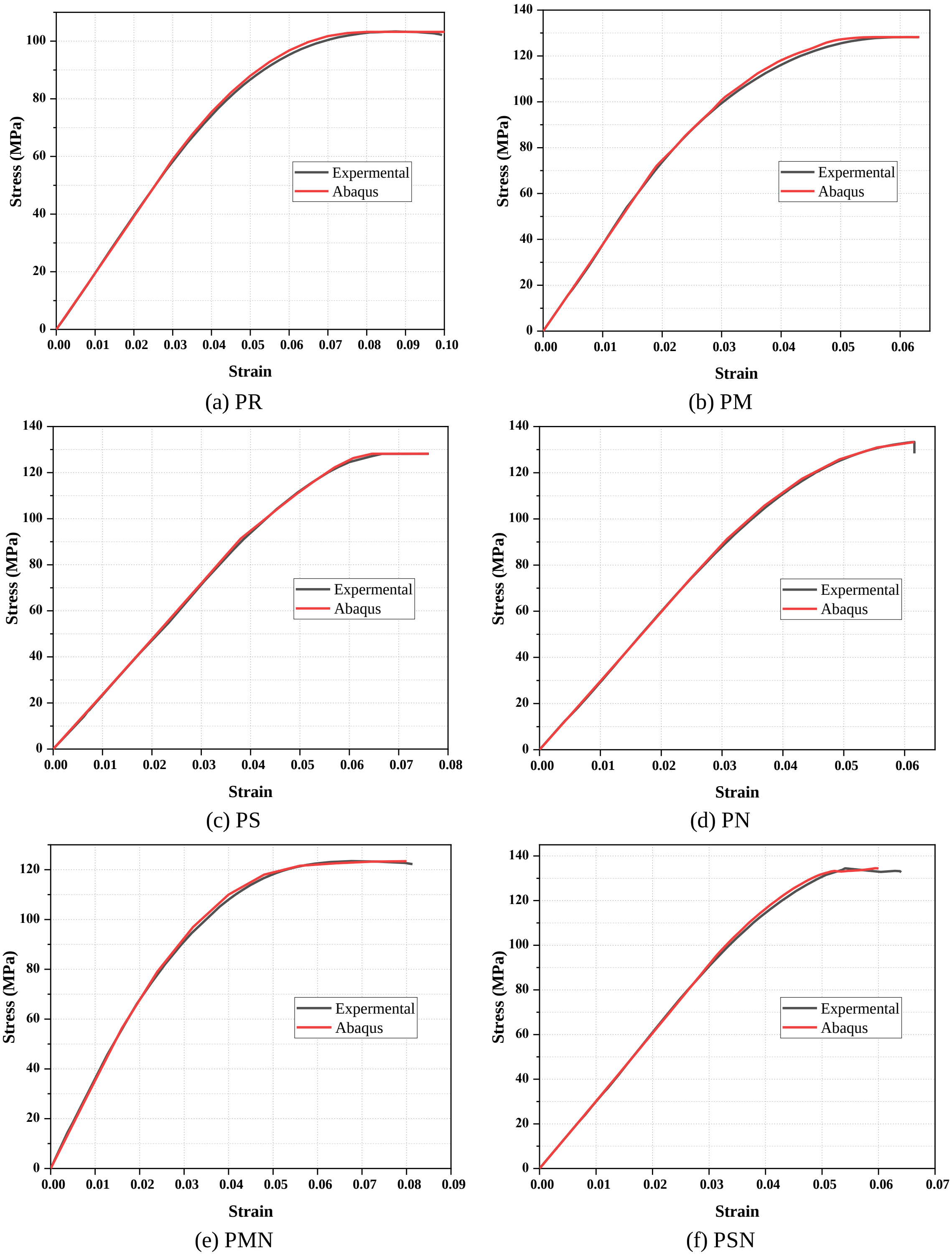
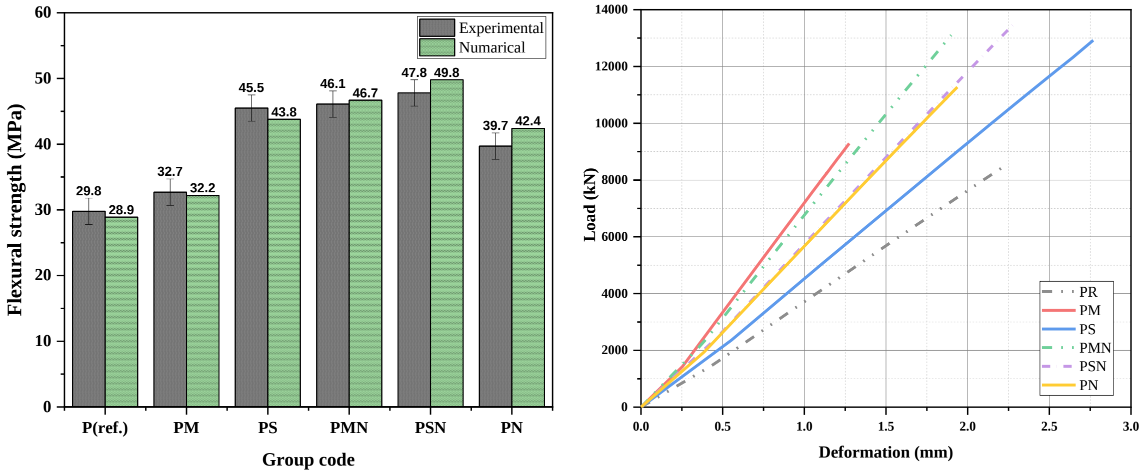
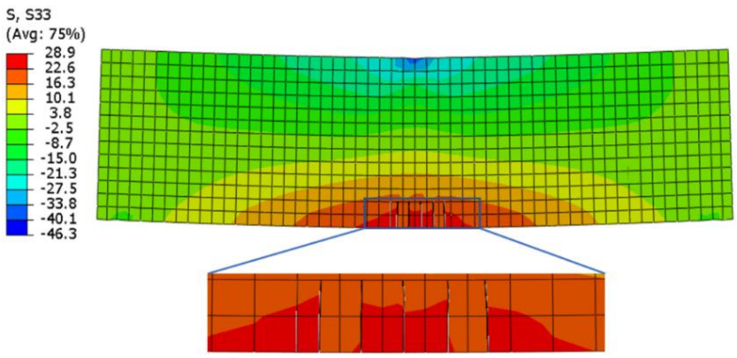


Fig. 9 Experimental and numerical stress-strain curves for neat/reinforced polymer composites

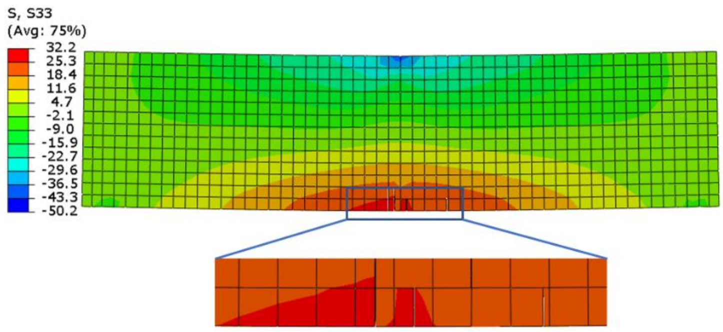


(a) (b)

Fig. 10 (a) experimental and FE flexural tensile strength results (b) load-deformation curves



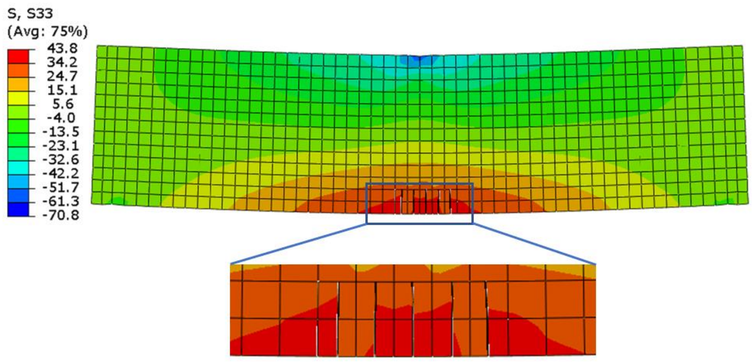
(a) PR



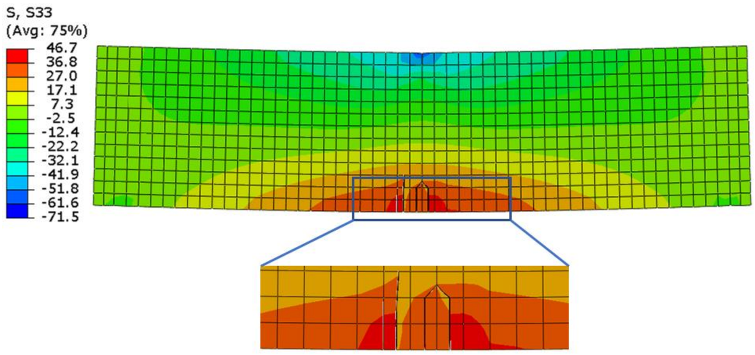
(b) PM

Fig. 11 Flexural test numerical model using XFEM

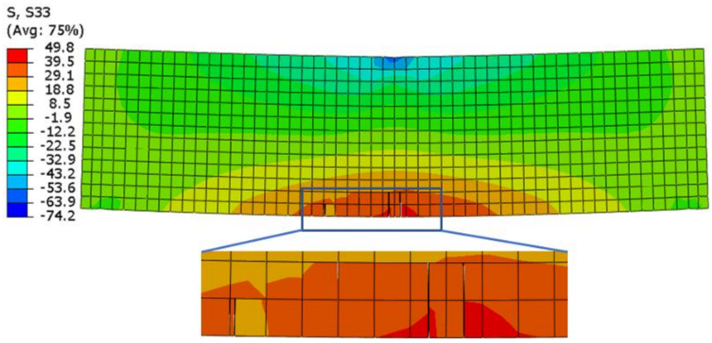
discontinuities in a FE by enriching degrees of freedom (DoF) using special displacement functions (Li *et al.* 2017). Fig. 11(a)-(f) shows the distributions of stresses of neat and reinforced polyester composites in the loading direction. Stress fields were uniform and homogeneous. The



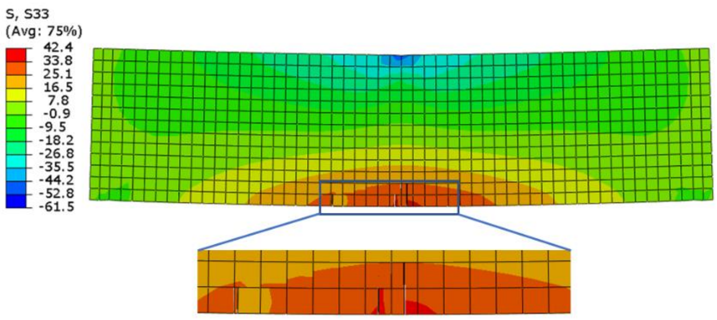
(c) PS



(d) PMN



(e) PSN



(f) PN

Fig. 11 Continued

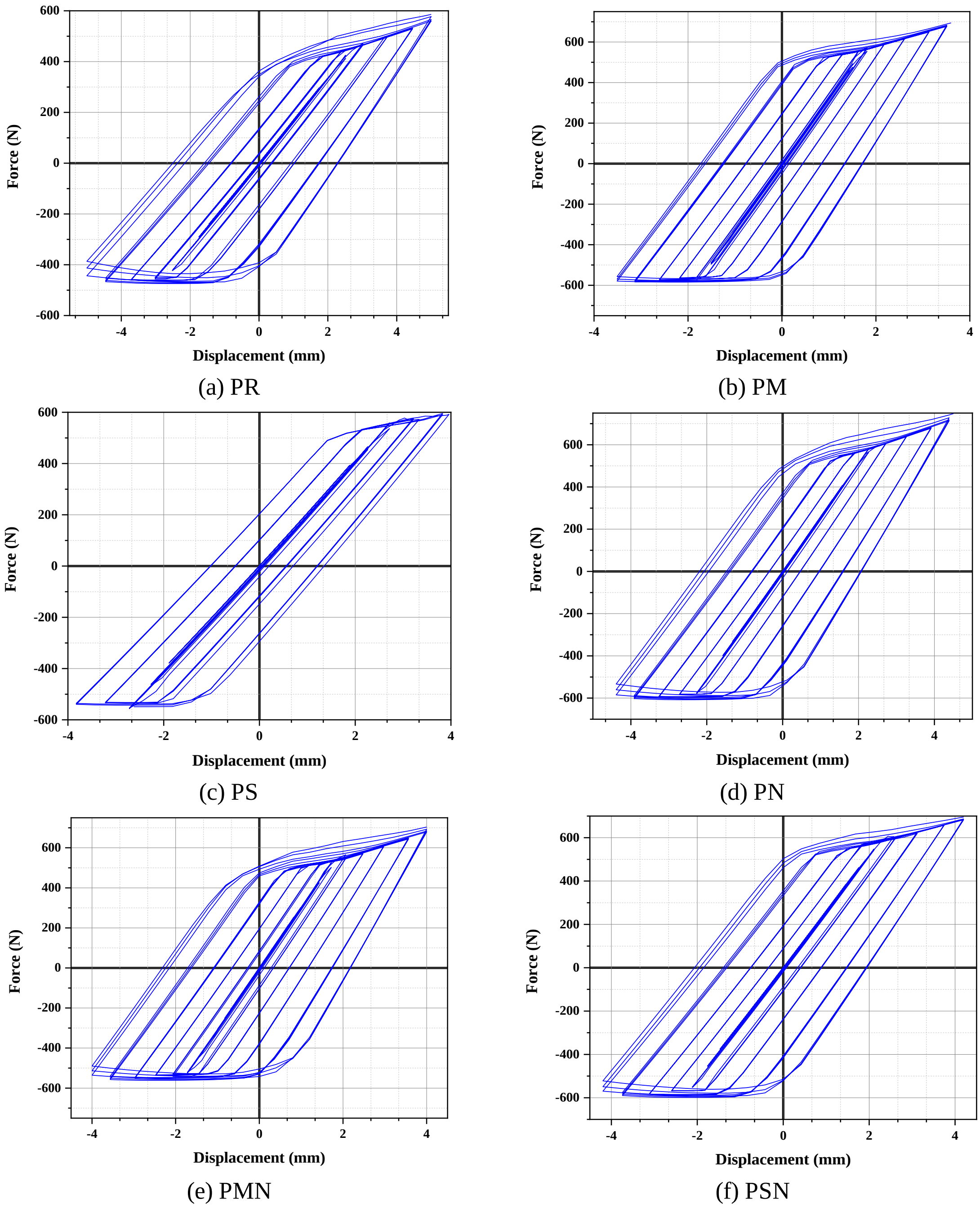


Fig. 12 Tension-compression hysteresis loops for polymer composites

maximum stress concentration for all groups was observed in the middle of the samples close to the crack zone. Cracks starting from the lower surface (tensile zone) and moving towards the upper surface (compression zone).

3.3 Hysteresis loops

A displacement control tension-compression loading was performed in ABAQUS software. Hysteresis loops were recorded in terms of displacement and reaction force at the sample base. The force-displacement hysteretic curves of the neat and reinforced polyester composite specimens are obtained and shown in Fig. 12(a)-(f). In general, all groups showed large deformations, implying more ductile behavior than the single silica fume reinforced group.

4. Conclusions

Reinforced polyester composite is a new structural material with better mechanical and durability performance in concrete repairs and bridge decks overlays. The compressive strength, flexural strength and tensile-compression fatigue behavior of polyester composites with different sizes of fillers were investigated experimentally and numerically, and the following results were obtained:

• Added silica-based fillers such as silica fume, marble powder or nano-silica improved the compressive strength of polyester composites by 32-38% and flexural tensile strength by 10-60% compared to pure polyester composite.

• The results of the FE model are in agreement with the experimental compression and flexural test results.

• ABAQUS was successfully coupled to predict the uniaxial tension-compression fatigue behavior of neat/reinforced polyester composite specimens.

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