



Polymer-Impregnated Cement Mortar: Effects of PEG, PAM, and PVA on Mechanical Properties

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ABSTRACT

The brittleness and porosity of cement mortar leads to low compressive, flexural, and tensile strengths and poor hardness, making it susceptible to environmental degradation. This study aimed to improve the mechanical and physical properties of cement mortar using a simple and cost-effective approach of impregnating pre-cured hardened mortar with polymers. Three polymers - polyethylene glycol (PEG), polyacrylamide (PAM), and polyvinyl alcohol (PVA) - were used for impregnation. The polymers were blended with a magnetic stirrer and the impregnation was performed via three methods: vacuum, ultrasound, and 24-hour immersion. The results showed significant improvements in mechanical and physical properties. PEG-impregnated samples exhibited the highest compressive strength (24.47 MPa), flexural strength (1.38 MPa), and splitting tensile strength (2.08 MPa) compared to reference samples with 17 MPa, 0.52 MPa, and 1.35 MPa respectively. PAM-impregnated cement mortar displayed the highest hardness value of 81 versus 70.08 for the reference sample. Optimal results were achieved via the vacuum method, with increases in bulk density. The polymer impregnation filled pores and improved bonding, enhancing the mechanical properties of the brittle cement mortar.

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1. Introduction

High-strength concrete is currently produced by adding chemicals, minerals and fibers. Because of these technological and financial advantages, concrete will continue to be the most common and necessary material in the construction sector for years to come [1, 2]. Although concrete is a widely used building material, it also has weaknesses, including porosity. For this reason, the material is being changed to improve its strength, durability, energy efficiency and other properties. Recent studies have focused on improving the pore system in cement concrete. Polymers have been used to improve the properties of cement concrete, and commercial interest in polymer-impregnated concrete (PIC) has increased significantly. Polymers in cement concrete can improve various properties such as strength and durability by strengthening the internal structure and various physical properties such as corrosion resistance and porosity. As PIC has a strong resistance to water penetration and prevents salt water from corroding the steel reinforcing bars, it is used on the surfaces of concrete bridges [3, 4].

Chen *et al.* experimentally investigated the influence of soaking time and polymerization temperature on the mechanical and physical properties of PIC. During impregnation, the soaking time was set to 4, 8, 12, 16, 20 and 24 hours and the polymerization temperature was set to 70°C, 80°C and 90°C for 0.5, 1, 2, 4, 6 and 12 hours, respectively. Methyl methacrylate (MMA) and benzoyl peroxide were impregnated into cylindrical concrete test specimens with a water/cement ratio of 0.45 and 0.65. The polymer load increased up to an exposure time of 12 hours. The ideal polymerization temperature for mix A (high cement concentration) was 70 °C and for mix B 80°C, based on compressive strength (CS) and surface absorption (low cement content). Compared to normal concrete, the PIC had significantly had lower surface absorption but higher strength and resistance. Polymethyl methacrylate was used to fill the micro- and mesopores of PIC samples and drastically reduced the total pore volume and maximum pore size, as shown by scanning electron microscopy (SEM) and maximum inspiratory pressure (MIP) measurements [5].

Wu *et al.* have investigated the effect of ultrasonic waves on increasing the effectiveness of monomer impregnation in hardened concrete without prior drying; low-intensity ultrasonic waves (20 kHz, 0.19–0.57 W/cm² relative to the concrete surface) were also used for subsequent in situ polymerization. Without removing free water from the sample, MMA can be impregnated into partially air-dried Portland cement concrete to a depth equivalent to approximately 80% of the depth of a corresponding previously oven-dried sample.

The relationship between the depth of impregnation and the following factors was investigated: Impregnation time, impregnation energy density, distance between the ultrasonic horn and the sample surface, and the applied DC voltage gradient. In this work, the experimental results are presented and an explanation for the ultrasonic amplification is proposed [6]. A review of previous research on PIM and PIC revealed that the in-situ polymerization technique is widely used for polymer impregnation of concrete. By applying the PIM technique, researchers hope to improve the properties of mortar, including its ductility, shrinkage resistance, flexural strength, compressive and splitting tensile strength, durability, and abrasion resistance.

In this study, a polymer impregnated mortar was produced using three different methods: (ultrasonic, vacuum, immersion in a polymer solution for 24 hours). To improve the mechanical and physical properties of the mortar for maintenance and restoration, polyacrylamides (PAM), polyethylene glycol (PEG) and polyvinyl alcohol (PVA) were used to impregnate the cement mortar. These types of polymers were also used because of their good compatibility with cement mortar and their excellent mechanical properties, which improve the resistance of the cement mortar samples.

2. Experimental Procedure

2.1 Materials

The cement used was ordinary Portland cement type 1 from Lafarge Company/Bazian, also known as Karasta. The cement was carefully stored in airtight plastic containers to protect it from external influences, especially moisture. Table 1 lists the chemical composition of the cement and Table 2 describes the physical properties of the cement. These properties are important determinants for the quality of the cement and its usability for different purposes. Natural sand from AL-Ukhaidir with a final grain size of 4.75 mm and a limited grain size was used for the entire experiment. Normal tap water was used for mixing and curing the concrete mixes throughout the experiment.

Table 1: Chemical composition of cement and weight percentages of oxides.

Composition	Abbreviation	Weight percentage%	Limits of (IQS NO.5/1984)
Lime	CaO	61.69	-
Silica	SiO ₂	18.91	-
Alumina	Al ₂ O ₃	3.74	-
Iron oxide	Fe ₂ O ₃	4.23	-
Sulphate	SO ₃	2.25	≤2.8
Magnesia	MgO	1.90	≤5%
Loss on Ignition	L.O.I	2.39	≤4%
Insoluble Residue	I.R	1.5	≤1.5%
Lime Saturation Factor	L.S.F	0.95	0.66-1.02

Table 2: Physical properties of cement.

Physical properties	Results	Limits of (IQS NO.5/1984)
Setting time (Vicat's method) (in min.): (Initial, final)	(2.05, 4.00)	≥1 h, ≤10.00 h
Soundness (autoclave method) %	0.12	≤0.8%
Compressive strength (70.7 mm cube) (N/mm ²) (for 3 days, for 7 days)	(20, 25)	≥15, ≥23
Specific gravity	3.15	-

The polymers used in this study included polyacrylamide, polyethylene glycol (PEG-20000), and polyvinyl alcohol (PVA). Polyacrylamide is a water-soluble polymer commonly employed as a thickening or flocculating agent in various industries due to its versatile properties [7]. Polyethylene glycol (PEG-20000) has the chemical formula $[(C_2H_4O)_nH_2O]$ and is a highly hydrophilic polymer that can dissolve in solvents such as acetone, alcohols, hydrocarbons, and water. The physical properties of PEG, including solubility, moisture absorption, vapor pressure, freezing point, and viscosity depend on its molecular weight. The PEG used had a specific gravity of 1.12 and a molecular weight of 20,000. Polyvinyl alcohol is a colorless, synthetic resin soluble in water. It is one of the few polymeric substances composed of diverse molecular units. The PVA polymer employed had a melting point of 200°C and a density of 1.19 g/cm³.

2.2 Preparation of Samples

Cube, rectangular, cylindrical, and disk samples were produced according to Table 3. Once the molds were ready and lubricated, a combination of cement and sand in a proportion of 1:3 was incorporated. Adding water until the cement content reached 0.45, the well-mixed dry components were stirred together. Next, the prepared molds were filled with the mixture and left to cure for 24 hours. For the hydration processes, the mixtures were removed from the molds and immersed in water for 28 days. The samples that were poured into the molds are shown in Fig. 1.

Table 3: Shapes and dimensions of the samples according to the standard specification.

The test	Shape	Dimension, mm	Specification
Compressive strength	Cubic	50×50×50	ASTM C 109/C109M-20b
Flexural strength	Prism	40×40×160	ASTM C 293/C293M -16
Splitting tensile	Cylinder	50×100	ASTM C 496/C496-17

The three types of polymers were used to produce the polymer solution: PEG, PAM and PVA. For the polyethylene glycol polymer, 10 g of polyethylene glycol was dissolved in 100 ml of water at room temperature and the dissolution process was conducted using a magnetic stirrer for 40-50 minutes. In polyvinyl alcohol polymer, 10 g of polymer was dissolved in 200 ml of water at a temperature of 70 °C for 60-70 minutes and the dissolution process was performed with a magnetic stirrer. For polyacrylamide polymer, 0.3 g of polymer was dissolved in 20 ml of water at room temperature for 30-40 minutes using a magnetic stirrer.

After the cement mortar samples had cured for 28 days, the samples were immersed in the polymer solution for 24 hours using three techniques: The vacuum technique used a vacuum apparatus consisting of a vacuum container and a rotating device. When the sample is immersed in the solution in the vacuum system for three hours, the compressive force resulting from the process causes the container to deflate and the polymer liquid to penetrate the sample, allowing the polymer solution to diffuse into the pores of the sample. An ultrasonic wave generator (Korea; model, LUC-410; 400 W) was used for the ultrasonic technique. The vibrations of the ultrasonic waves push the polymer solution into the sample which has been placed in the device for 3 hours, facilitating the distribution of the polymer solution in the sample. Finally, during Impregnation with PAM, the sample was immersed in the polymer solution for 24 hours applying no external pressure to the polymer solution. The samples were tested after performing the impregnation methods.



Figure 1: Different testing samples before and after setting. a- flexural strength samples, b-compressive strength samples, c-splitting tensile strength samples, and d- samples after setting.

2.3 Structural and Mechanical Properties

Under ASTM C 109/C109M-20b, cubic samples were evaluated under a compressive force [8]. The test was performed with a control machine type 065-10019/B with a load capacity of 250 kN. By dividing the maximum force applied to the samples during the test to cause ultimate failure by the average cross-sectional area of the samples, the compressive strength (CS) of the sample was calculated. You can calculate it using Eq. (1) below.

$$CS = \frac{F}{A} \quad (1)$$

Where, F is the applied load (N), and A is the cross - sectional area (mm²).

Prism samples measuring were put through a flexural compliance test under ASTM C 293/C293M-16 specifications [9]. A controlled machine with a load capacity of 250KN, model 065-10019/B, was used to conduct the test. The prism was put through the three-point test, which involved center-point loading. The Eq. (2) below can determine flexural strength (FS):

$$FS = \frac{3 \times F \times L}{2 \times b \times d^2} \quad (2)$$

Where L is the distance between two supports (mm), b is Width of the prism sample (mm), and d is Thickness of prism sample (mm). A cylindrical specimen was positioned with its horizontal axis between two supports of the tensile testing machine, which has a load capacity of (250 KN). The tests were performed under ASTM C 496/C496-17 for split tensile strength [10]. There was a failure after the production of cylinder specimens and loading. The Eq. (3) below was used to determine the values for split tensile strength (STS):

$$STS = \frac{2 \times F}{\pi \times D \times L} \quad (3)$$

Where, F is the Maximum Load (N), D is the diameter of the sample (mm), and L is the length of the sample (mm).

The microstructure and interfacial bonding between the polymers and cement mortar were examined using field emission scanning electron microscopy (FESEM). Small pieces of the cured polymer-impregnated cement

mortar samples were mounted on aluminum stubs using carbon tape. To enhance conductivity, a thin film of gold was sputter-coated on the samples for 90 seconds before placing them in the FESEM chamber. The analysis was performed under high vacuum conditions at an accelerating voltage of 10 kV using an FESEM (Zeiss Sigma 300- HV). Different magnifications ranging from 500x to 10,000x were used to study the dispersion of polymers within the mortar matrix and their interfacial interaction.

The hardness of the cured polymer-impregnated cement mortar samples was evaluated using a Shore D durometer. The durometer works by pressing the indenter foot firmly onto the sample to be tested. The pressure causes the spring inside to deflect and the hardness value is measured on the scale. For each cement mortar composition, 5 readings were taken on different areas of each sample. The tip of the durometer was held vertically on the flat surface of the sample and the presser foot was allowed to settle for 1-2 seconds before noting down the hardness number. The average of 5 readings was reported as the Shore D hardness of that specimen.

3. Results and Discussion

3.1 Compressive Strength

The comparison of the measured CS of polymer-impregnated samples with three different polymers (PEG, PAM and PVA) with the reference sample is shown in Fig. 2. Each sample impregnated with polymers showed an increase in CS for each impregnation method. This result is because the compressive strength of the samples increases when the interstices in the cement mortar are filled with polymers [11]. Because of the higher molecular weight of PEG compared to PAM and PVA, the sample impregnated with a PEG polymer using the vacuum method exhibited the highest CS (24.47 MPa). In contrast, 17.03 MPa was measured for the reference sample [12]. It was found that the compressive strength increased only slightly, and that the addition of polymer-cement mortar improved the compressive strength. This result is consistent with a study by Dębska *et al.* [13]. If the PAM-impregnated samples are compared with the reference samples with CS values (17.03 MPa), the compressive strength increases significantly. The sample subjected to the vacuum technique yielded the highest PAM-PIM value (24 MPa) [1, 14, 15].

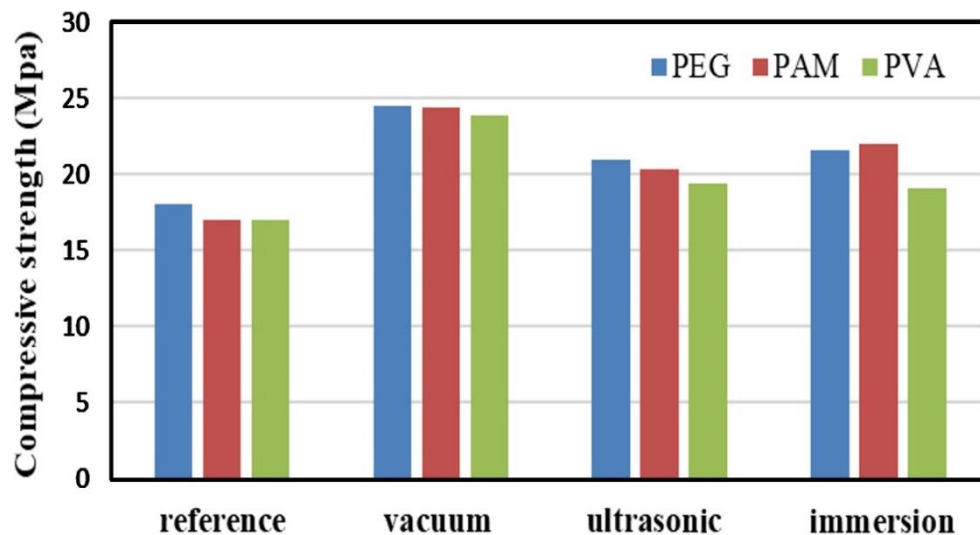


Figure 2: Compressive strength results of PIM.

PVA-impregnated samples, which also showed a significant increase in the compressive strength of the sample after impregnation with the PVA polymer, had a compressive strength value of 23.87 MPa, which was significantly higher than the reference sample's value of 17.03 MPa. In contrast to the reference sample, each sample was subjected to a compression test at the same time and at the same temperature.

As the polymers filled the spaces between the pores of the mortar sample, the polymer-impregnated samples exhibited higher compressive strength. In agreement with the results of a previous study, the compressive strength increased as the penetration of the polymers decreased [16]. When the polymer filled the pores in the cement mortar, which was the main reason for the sample's brittleness and fragility, the strength of the sample increased. The behavior of the polymer and its advantageous compressive strength properties are responsible for this improvement in compressive strength values [13, 17].

By utilizing the vacuum method, the sample impregnated with PEG polymer achieved the highest CS value (24.47 MPa). This is because of the pressure exerted on the polymer liquid forcing it into the specimens and the spreading of the polymer in the internal pores of the cement mortar specimen. It was found that the results got with ultrasonic impregnation were lower than those obtained with impregnation without external action. This could be because of the heat emitted by the ultrasonic wave of the device, which causes an increase in the polymerization speed, or to the fact that the polymer was not homogeneously distributed in the internal pores of the cement mortar sample. In addition, the bond strength between the polymer and the mortar affected the compressive strength, with the C.S. increasing with the strength of the bond and a high load being required to break the bonds [18–20].

3.2 Flexural Strength

Fig. 3 shows the results of the flexural strength test, from which the results show all samples impregnated with three different polymers (PEG, PAM, PVA) have a higher flexural strength compared to the reference sample than the samples impregnated with polymers. The sample impregnated with PAM in a vacuum process showed a maximum value of 2.49 MPa. The flexural strength was improved by dip impregnation and ultrasonic testing. In contrast, the flexural strength of the cement mortar sample was 0.52 MPa [21–23]. All specimens impregnated with different techniques exhibit a remarkable increase in flexural strength, as shown in the identical figure with the PEG-PIM specimens. The flexural strength of the sample impregnated with the vacuum method, which reached 2.491 MPa, was significantly higher than the flexural strength of the reference sample (0.52 MPa). The increase was caused by the polymer liquid applying additional pressure on the mortar sample while it was under vacuum [24, 25].

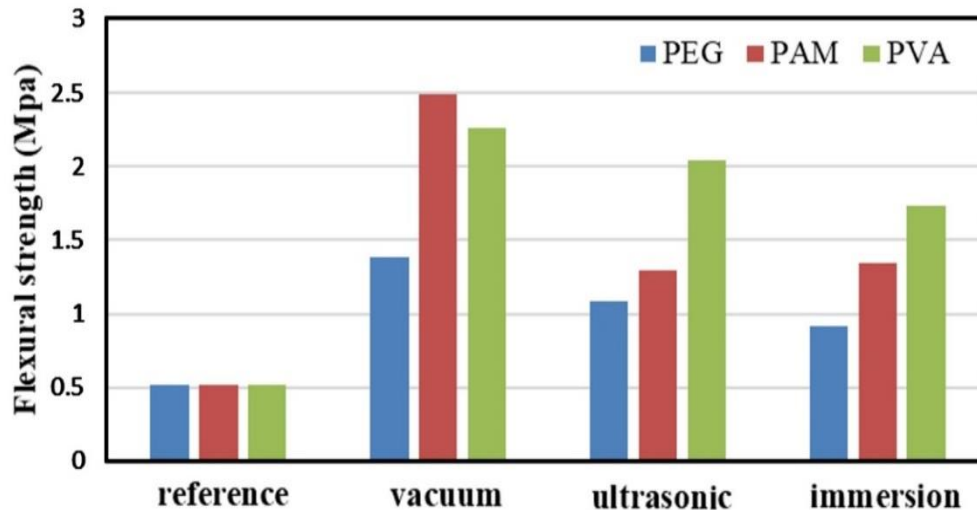


Figure 3: Flexural strength results of PIM.

The comparison of the results of the development of flexural strength of polyvinyl alcohol impregnated specimens with the reference specimen shows all specimens were subjected to flexural testing at the same time and temperature, because the results show a significant improvement in the flexural strength of the PVA polymer impregnated specimens impregnated with three impregnation methods (vacuum, ultrasonic and immersion for 24 hours) compared to the reference specimen, where the flexural strength depends on the amount of polymer in the

cement sample. Compared to the reference sample, whose flexural strength was 0.52 MPa, the flexural strength measured for this approach was 2.26 MPa. The flexural strength increased significantly with ultrasonic impregnation and 24-hour immersion in PVA, reaching 2.04 and 1.73 MPa, respectively. The presence of the polymer causes the pores to fill with the polymer, increasing the bond strength with the specimen and covering the mortar interface, which explains the increase in flexural strength [16].

Brittleness is influenced by flexural strength, leading to the polymer-impregnated specimens exhibiting lower brittleness compared to the reference specimens. The results of a previous study showed that the polymer and mortared cement formed robust bonds that increased flexural strength [26]. The increase in flexural strength correlated with the extent to which the polymer was fully absorbed, dispersed, and impregnated into the cement mortar samples. As the vacuum technique accelerates the impregnation process by squeezing out the polymer liquid and forcing it into the sample, the impregnated sample with the highest flexural strength was produced.

3.3 Split Tensile Strength

Fig. 4 shows a comparison between the results of the increase in tensile strength of the samples impregnated with polymers and the reference sample. Namely, the results show a significant increase in the splitting tensile strength of the polymer-impregnated sample for the three impregnation methods (vacuum, ultrasonic and 24-hour immersion) compared to the reference sample. Fig. 5 shows the sample during the splitting tensile strength test, where a continuous pressure applied to the cylindrical sample at a constant speed of 0.5 m/s until the final fracture. A regular fracture was observed when the sample was split in the middle. Each sample impregnated with the polymer showed a higher splitting tensile strength compared to the reference sample. The sample impregnated with the polymer using the vacuum method showed the highest splitting tensile strength compared to the samples impregnated using other methods, which can be attributed to the influence of pressure on the impregnation. The vacuum technique and the PEG polymer resulted in an achieved splitting tensile strength of 2.082 MPa, whereas the reference sample had a strength of 1.346 MPa. A splitting tensile strength of 1.890 and 1.722 MPa was achieved with ultrasonic and a 24-hour immersion in PEG [15]. When comparing the splitting tensile strength of a mortar sample impregnated with PAM polymer to a reference mortar sample, it is evident from the same figure that the highest value (4.111 MPa) was achieved with the polymer-impregnated sample after 24-hour immersion, surpassing the value of the reference sample (2.617 MPa). In the case of the sample impregnated using ultrasonic and vacuum techniques, the values were 3.675 and 3.536 MPa, respectively. Information on the process from crack propagation to splitting of the sample was obtained through the splitting tensile strength test [14, 27].

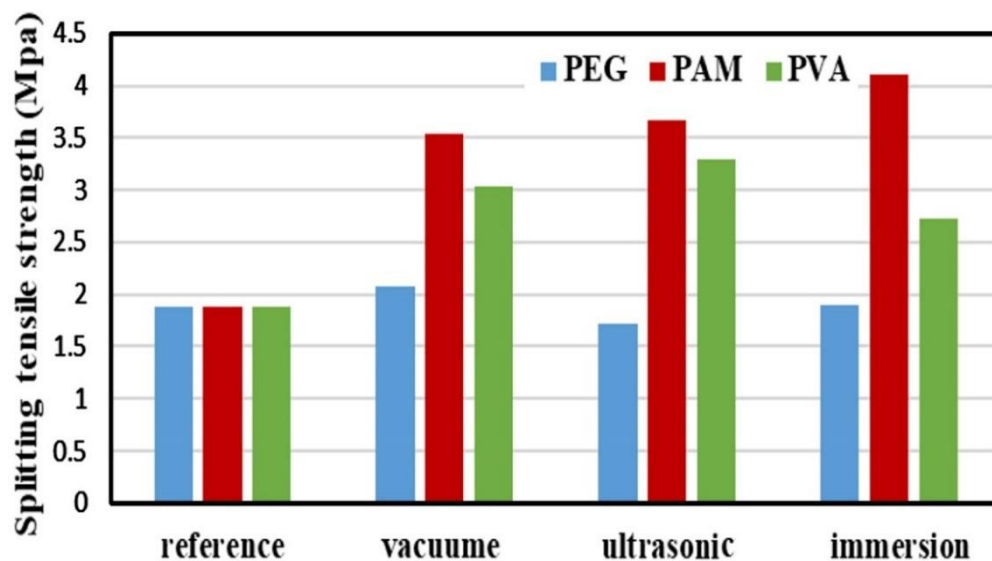


Figure 4: Tensile strength of PIM compared with the reference.

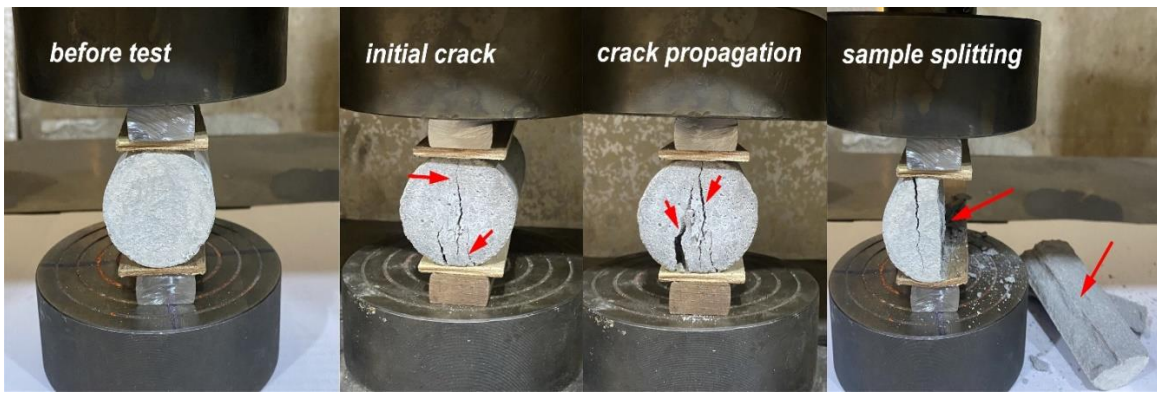


Figure 5: Crack propagation under splitting tensile test.

Each sample underwent the splitting tensile test at the same temperature and duration. All samples impregnated with polyvinyl alcohol and produced with all techniques showed a higher splitting tensile strength compared to the reference sample. The sample impregnated with the ultrasonic showed the greatest increase, suggesting that this technique caused the highest polymer impregnation and that the reason for this may have been polymer diffusion within the pre-existing pores. The cement mortar sample is consolidated because the polymer covers most of the pores, resulting in friability and weakness. In contrast, immersion in a polyvinyl alcohol solution for 24 hours also resulted in a good increase in splitting tensile strength with a value of 2.73 MPa [14, 27].

Cement mortar samples that were impregnated with ultrasound and vacuum technology yielded the best results. The reason for this is the increasing spread of the polymer in the pores inside the sample because of the ultrasonic and the vacuum pressure, whereby the diffusion of the polymer liquid into the sample increased with increasing vacuum pressure. As the polymer filled the pores, the splitting tensile strength of the sample impregnated with the polymer increased compared to the reference sample, enhancing the strength and durability of the cement mortar [14, 27].

3.4 Shore Hardness

Fig. 6 compares the Shore D hardness of a cement mortar sample with a cement mortar sample saturated with three different polymers (PEG, PAM and PVA). The results show that each cement mortar sample impregnated with polymers outperforms the reference sample in terms of hardness resistance. The hardness of the cement mortar samples was achieved by adding a polymer with good hardness properties, which also made the samples more scratch resistant. With a hardness value of 87.9, the PAM-impregnated cement mortar had the highest hardness value. Compared with the reference sample, which had a hardness value of 70.08, the ultrasonic method produced a hardness value of 80.3, while the dip impregnation only produced a hardness value of 79. When the cement mortar was impregnated with PEG, the hardness value also improved. Among the methods used, the vacuum method yielded the highest hardness value of 81. Comparatively, the ultrasonic method resulted in a hardness value of 73, while the immersion impregnation method only produced a hardness value of 76 when compared to the reference sample, which had a hardness value of 70.08. By impregnating the cement mortar with PVA, both the hardness resistance and the hardness value were increased. The ultrasonic method gave the highest hardness value of 81, while the vacuum method gave a hardness value of 77.6 and the immersion method only gave a hardness value of 77.5 compared to the sample. The hardness value of the benchmark was 70.08.

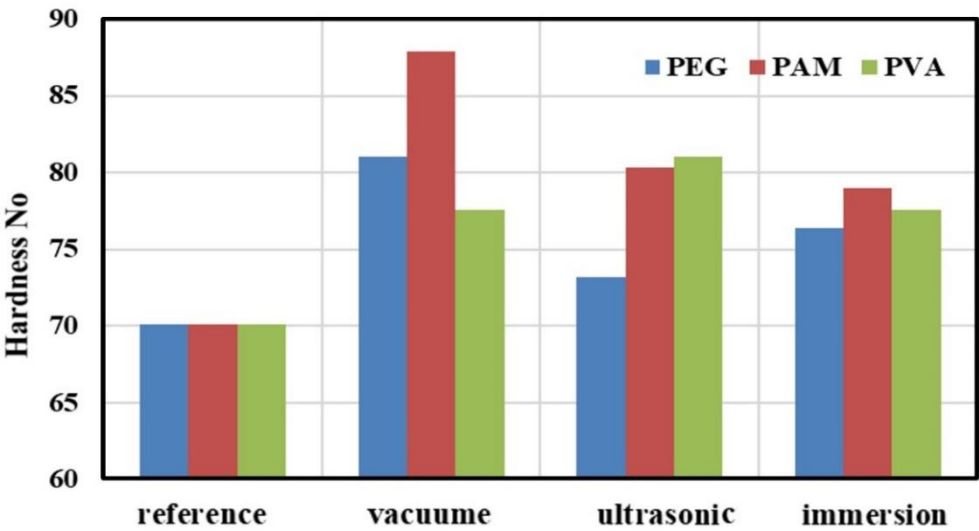


Figure 6: Hardness (Shore D) of PIM compared with the reference sample.

3.5 Filed Emission Scanning Electron Microscopy (FESEM)

Fig. 7 shows SEM images of cement mortar samples without polymer impregnation. Details of the composition of cement, sand, voids and pores can be seen in the sample. Chemical reactions between the different components of the sample can also be seen, as the images show the homogeneity of the cement mixture in the cement mortar sample. The Ettringite, $\text{Ca}(\text{OH})_2$ and C-S-H grew with age in the ordinary Portland cement mortar and by the twenty-eighth day of aging, the microstructure and texture of the slurry became more complicated.

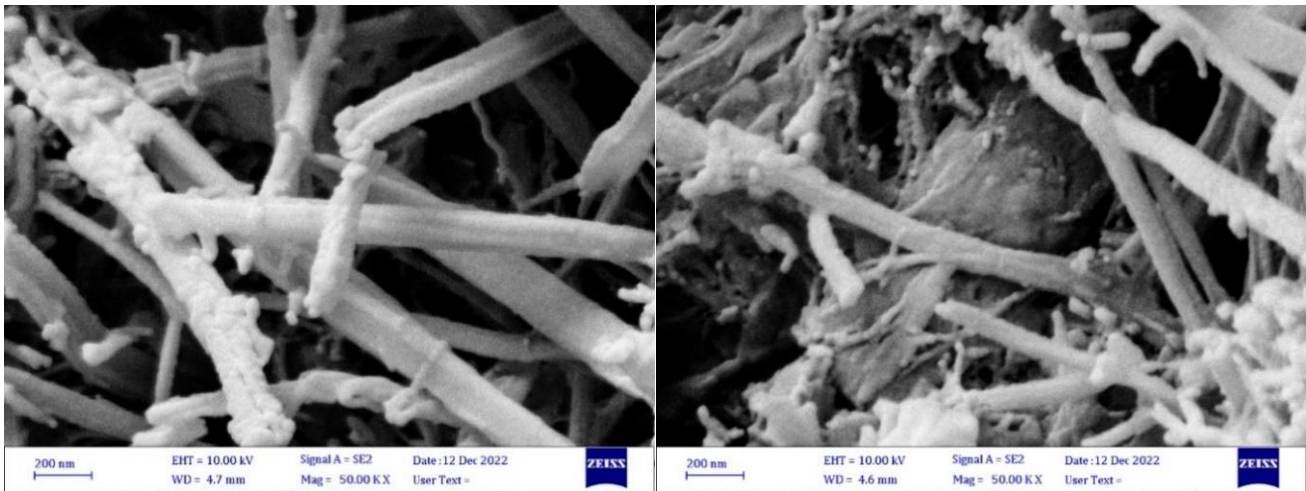


Figure 7: The microstructure image of the cement mortar sample.

The image captured by scanning electron microscopy (SEM) in Fig. 8 shows the fracture surface of the samples impregnated with polymers. The polymer in the PIM sample is dispersed, as showed by the homogeneous distribution seen in the SEM diagrams. Most of the pores are covered with the polymer and are spread out across the surface. The scanning electron microscope images show an agglomeration of the polymer on the pore surfaces and these agglomerations appear as a network on the fracture surfaces as they are pores.

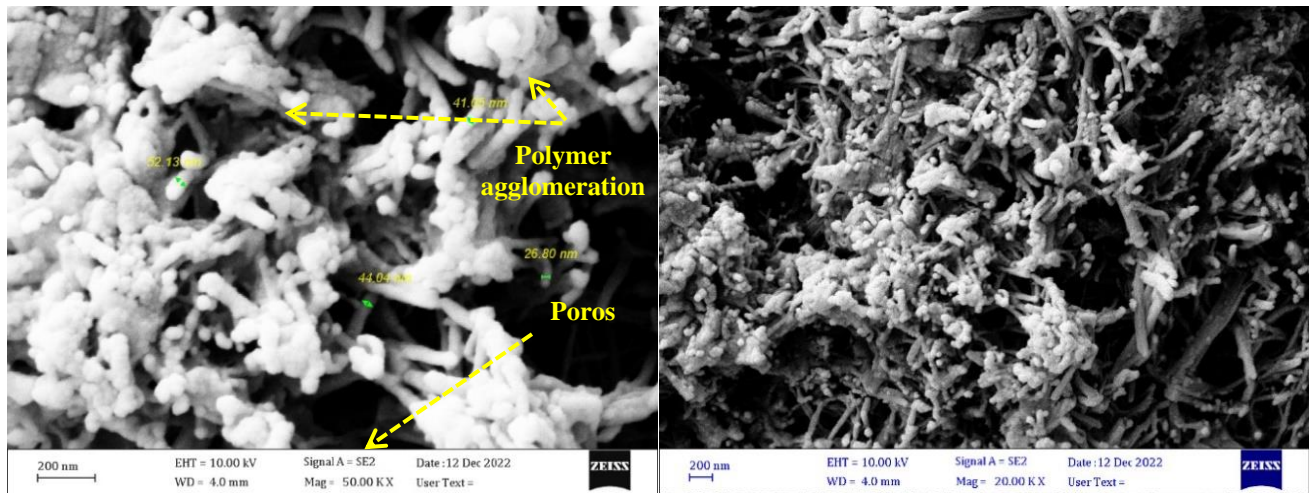


Figure 8: The microstructure image of the PIM sample.

4. Conclusions

The pre-polymer can be added to the cement mortar sample by impregnation procedures using three different methods, all of which gave good results, with vacuum impregnation being the best method. Three different polymers were used to impregnate the cement mortar sample: PEG, PAM, and PVA. PEG yielded the best results. A significant increase in compressive strength was observed, with the highest increase of 43.94% obtained in the cement mortar sample impregnated with PEG using the vacuum method. The highest deflection in the flexural test was found to be 292.3% in the cement mortar sample impregnated with PVA using the ultrasonic method. The splitting tensile strength test revealed a substantial improvement in splitting tensile strength, with the PAM polymer impregnated sample and the dipping technique producing the highest value in the results. By utilizing the vacuum technique, the cement mortar sample impregnated with PAM attained the highest hardness.

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Conflict of Interest

The authors declare that they have no conflict of interest.

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