International Journal of Nanoelectronics and Materials
Volume 16, No. 4, October 2023 [865-882]

The influence of post-curing periods on polymer-mortar composites in corrosive media

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Received 9 November 2022, Revised 1 February 2023, Accepted 6 March 2023

ABSTRACT

Petroleum tanks and pipes made of steel or concrete suffer from the interaction between the petroleum products and the materials of the tank or pipe. This work aims to overcome this problem by adding polymer to the cement mortar and post cure it at the proper period to modify its behavior when encountering petroleum products. Unsaturated polyester was selected to prepare polymer cement composites. Four sets of polymer cement composites were prepared. Two sets were cured at room temperature, but one was kept without immersing in any solution. The other sets were post-cured at 50ºC for two and four hours. Six groups of polymer cement composite specimens were immersed in a solution. The solutions were water, 1N and 2N NaCl, kerosene, benzene, and oil engine. The hardness and compressive strength were evaluated after 30 days. The hardness of the specimens was altered according to the solution immersed. The best water absorption and compressive strength results were obtained two hours post-curing specimens, whatever the solution was immersed in. The corrosion rate for all specimens in every solution was low. Again, the lowest corrosion rate was obtained after two hours of post-curing specimens, which indicates that the addition of polymer and proper post curing will improve the chemical resistance of the cement-sand mixture.

Keywords: Composite materials; polymer-mortar; post-curing; hardness; compressive strength.

1. INTRODUCTION

The material's durability in corrosive environments and concerns about property degradation may have an impact on the buildings' service life, necessitating a full analysis of the situation [1]. Corrosion and deterioration of the characteristics of tanks and pipelines are common in the petroleum industry. Crude oil is an organic material comprised entirely of carbon and hydrogen atoms that link together in different ways, with additional specific components such as sulfur, nitrogen, and oxygen atoms found in crude oil and its discharges. The sulfur concentration varies across all crude oil types, often in the compound form [2]. Concrete has been used to store petroleum products but attempts to use the material to build crude oil tanks were ineffective in the beginning. Oil leaking via the joints has been identified as a serious issue [3, 4]. Concrete tanks have been utilized to store crude oil and petrochemical products underground, aboveground, floating, and offshore in recent years. Concrete's reliability as a building material has led to its usage in constructing storage tanks, based on a reasonable understanding of some of its essential qualities. However, it is widely acknowledged that little is known about the nature of this material's interaction with the diverse contents of these tanks. The permeability of concrete is mainly determined by its susceptibility to external assault [1, 2, 5], which is why most writers consider this attribute a quantitative indicator of concrete durability. The pore structure, instead of just the porosity of a mature normal-weight concrete, has been proven to affect permeability. It is also influenced by cracks and microcracks that might have developed

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due to faulty casting and curing techniques, ambient temperature, and shrinkage stresses [6]. Traditional steel pipes utilized in the petroleum industry have significant maintenance costs, corrosion, and shorter lifecycles. The entire yearly cost of corrosion in the oil and gas sector is significant, with pipeline and facility costs, downhole tubing fees, and capital expenditures for corrosion control absorbing capital expenditures [7].

The composite pipe is predicted to significantly minimize economic losses due to corrosion and high maintenance costs while opening up new investment options. A composite is made by combining two or more components to produce a new material that is stronger, lighter (in terms of comparable weight), and simpler to work with than separate elements such as metal and plastic [8, 9].Polymer modified concrete or mortar is a composite material made up of two solid phases: aggregates spread throughout the material and a binder made up of cementation and polymer phases.[10, 11] Cement polymer materials have been explored for use as a building material, promising greater strength, water tightness, and durability than traditional Portland cement concrete. In order to make polymer-cement composites, chemicals must be introduced into the concrete pores. The qualities of composite materials are determined not only by their elements but also by a synergistic effect [12]. Slow hardening, weak tensile strength, and significant drying shrinkage are some of the downsides of cement mortar and concrete. Many efforts to employ polymers to mitigate these drawbacks have been attempted.

Polymer-modified mortar (or polymer-cement) is one such effort, which is formed by mixing polymer additives such latexes, redispersible polymer powders, water-soluble polymers, liquid resins, and monomers with regular cement mortar. Several polymers with low glass transition temperature (Tg) may be used to make highly flexible polymer-modified cement (mortars) [13]. The flexibility of these materials, which allows waterproofing of concrete buildings with “moving cracks,” is the main reason. Furthermore, compared to traditional portland cement mortars or concrete, these materials have a much superior chemical resistance (even in acidic conditions) due to their high polymer content [14]. Mirza et al. [15] carried out a comprehensive experimental investigation to assess the strength, durability, and binding strength of two polymers (styrene butadiene rubber (SBR) and acrylics). The binding strength of polymer-cement mortars PCMs including SBR was found to be greater than that of PCMs containing acrylics, according to the researchers. In contrast, Al-Zahrani et al.[16] found no substantial variations in mechanical characteristics and penetration resistance between PCMs and standard cement mortar depending on test results and comparisons. When Aggarwal et al. [17] evaluated two PCMs using epoxy emulsion and acrylic emulsion, they found that the epoxy emulsion-based PCM had greater strength and durability. The influence of climatic variables on the adhesive strength and deformation of different PCMs was examined by Maranho and John [18]. They demonstrated that stable laboratory settings were preferable to outdoor exposure when illustrating the impact of test variables. Ariffin et al. [19] found that epoxy replacement ratios more than 10% decreased the strength of epoxy-modified cement mortar in an experimental study focusing on cement mortar strength. Li et al.[20] studied the effects of three distinct polymers (SBR, styrene-acrylic ester (SAE), and polyacrylic ester (PAE)) on the mechanical properties and durability performance of PCMs. SBR PCMs outperformed SAE or PAE PCMs in terms of strength, weight loss, and chloride ion penetration resistance. As a result, a safe storage system and technique for liquid goods that may become the target of assaults are needed. This may be accomplished by using the appropriate building material to guarantee that the desired durability, security, and economic advantages are realized. Furthermore, the ability to build buried tanks and increase strength without sacrificing structural flexibility may be acquired compared to steel tanks. The novelty of this work is to develop a polymer-mortar composite material without water that can survive corrosive media created by petroleum products and the surrounding environment to use as tanks or pipes that withstand these conditions. Unsaturated polyester was selected as the polymer as a matrix added to the cement-sand. Cement sand was considered reinforcement, used without water to prevent segregation. This composite was immersed in different solutions to evaluate its ability to resist the action of these solutions. The solutions selected were water and salt (NaCl as salt) as aqueous solutions encountered by the environment. Kerosene, benzene, and oil engines were chosen as petroleum (oil) products experienced when using these products as pipes or tanks.

2. EXPERIMENTAL DETAILS

2.1 The Materials

The flexibility and lightweight of the polymer-cement make it suitable for manufacturing tanks, pipes or even just coating the tank or pipes with it; to develop its behavior and protect it against any corrosive media. The research starts with materials preparation and ends with testing the preparing samples.

1- Ordinary Portland cement was kept in a dry place to eliminate the effect of humidity that could spoil the cement. The chemical and physical characteristics of cement are given in Table 1, which confirms the IRG "Iraqi Reference Guide" indicative number (198), the "Ministry of Planning/Central Agency for Standardization", and "Quality control Manual 198/1990" [21].

Table 1: Cement's physical and chemical characteristic.

Composition			Physical properties		
Item	**Content%**	**Spec.Limit**	**Items**	**Test result**	**Spec.Limit**
Al_2O_3	4.34	-	AutoclaveExp	0.24	0.8%
SiO_2	20.66	-	Fineness (m^2/kg)	310	230
Fe_2O_3	3.40	-	Compressive Strength (MPa)		
CaO	63.71	-			
MgO	2.07	5.0 max	3days age	17.2	15.0
SO_3	1.17	2.8max	7days age	26.0	23.0
LOI.	2.52	4.0 max	Time of setting		
I.R.	10.3	1.5	Initial (min)	75min.	45
			Final (hour)	4.15	10 max

2- According to "Iraqi Standard Specification No.45/1984", the sand was employed as fine aggregate, which was inside zone 2. Table 2 shows the gradation of fine aggregates.

Table 2 Grading of fine aggregate

Sieve size (mm)	%wt. Passing	Spec. Limit
4.75	93.2	90 to100
2.36	84.2	75 to 100
1.18	68.0	55 to 90
0.60	37.8	35 to 59
0.30	19.6	8 to 30
0.15	8.8	0 to 10
Percentage of salts%	0.4	≤0.5

3- Unsaturated polyester resin.
The most prevalent resins used in the composites sector are unsaturated polyester (UP) resins. The electrical, mechanical, and chemical properties of unsaturated polyester resin

(UP) are excellent. Table 3 shows the mechanical characteristics of polyester in general. UP "Unsaturated polyester" resins provide excellent chemical resistance. These resins perform well in weak alkalis and much better in mild acids. The polymer utilized in this project was unsaturated polyester, which is made up of the following ingredients:

a- The resin is the Unsaturated polyester (liquid).
b- The accelerator is Cobalt Naphthalate (liquid).
c- The hardener was Methyl Ethyl Ketone Peroxide (MEKP) (liquid).

Methyl Ethyl Ketone Peroxide (MEKP) is an organic peroxide which is colorless oily to yellow liquid with a significant odor. It has the formula of $(CH_3)\ (C_2H_5)\ C(O_2H)]_2O_2$ and it is used as a curing agent in polyester resins. It occurs commercially as a solution of Methyl Ethyl Ketone Peroxide (60%) and diluent (40%) to prevent explosions. Cobalt Naphthalate ($C_{22}H_{16}CoO_4$) is an additive used for curing unsaturated polyester resins at ambient temperature curing (not exceed >80°C).

Table 3 Properties of polyester resin

property	value
Density	1.15 g/cm^3
Flexural strength	176 MPa
Tensile strength	91.5 MPa
Flexural modulus	7.38 MPa
Tensile modulus	9.3 MPa

4- Petroleum products(oil products)

Kerosene and benzene are liquids derived from petroleum which are characterized as clear, colorless and low viscosity liquids. They are both hydrocarbons but kerosene chemical composition is fairly complex than benzene. Kerosene consists of paraffins, naphthenes, and aromatic hydrocarbons; while benzene contains only carbon and hydrogen atoms besides is considered a flammable liquid with a sweet smell unlike kerosene. Engine oils are composed of petroleum-based hydrocarbons, polyalphaolefins (PAO) blended together, or mixed in various proportions, sometimes with esters (up to 20% by weight).

2.2 Specimens Preparation

Four sets of polymer cement-sand composites were prepared. Two sets were cured at room temperature. The other two sets were cured at 50ºC for two and four hours. A temperature of 50ºC was suggested to accommodate the high-temperature climate. After curing, each specimen was weighed using a high accuracy balance. Six solutions were prepared to resemble the corrosive media encountered by the petroleum products and environment. The selected petroleum products were kerosene, benzene, and engine oil. The other solutions encountered by soil and environment were water and NaCl(sodium chloride) solution. This study selected tap water and NaCl solution with 1N and 2N normality. Each of the three sets was immersed in the six solutions separately, and the fourth group that cured at room temperature was left without immersing. After 30 days of immersion, each specimen was weighed.

The cement and sand (1:1 ratio) were manually dry mixed for 2 to 3 minutes to ensure a good homogeneity and distribution in the polymer cement composites. The unsaturated polyester resin was mixed with 0.05% accelerator (Cobalt Naphthalate), which gave it a pink color after it was transparent, then 2% hardener (MEKP) was mixed with them. Each accelerator and hardener should add in these specific percentages to keep the reaction under control because it is an exothermic reaction and prevents any internal stresses or excessive bubbles. After the polymer's homogeneity, the cement-sand mixture was gradually added to the unsaturated polyester. The percentage of adding polyester to the cement-sand mixture (1:1) was 40:60. Thoroughly manual mixing for 10 min is recommended to achieve the best distribution in the formed composites. These formed composites were molded layer by layer in a cylindrical 2x4 cm metal mold for good compaction. A lubricant layer was applied on the inner side of the mold to facilitate the de-molding of the composite specimens. The unsaturated polyester was added without adding any percentage of water to the cement-sand mixture. The suggestion of removing water from the mixture of composites is to acquire the best homogeneity with minimal pores. The existence of two matrices (the cement-water and polymer) is challenging to control the uniformity and homogeneity since the unsaturated polyester does not react with water, which causes segregation by unreacted parts.

On the other hand, the presence of polyester will hinder the reaction between cement and water. Besides, water leaves pores after evaporation which will cause holes to penetrate any liquid. Therefore, the cement in this composite is considered a reinforcing material, not a matrix. The formed composites were allowed to cure at room temperature for 24 hours to ensure complete hardening. The specimens were de-moulded after curing at room temperature, as shown in Figure (1). The specimens were divided into four groups. Two groups were cured at ambient temperature, while the other two groups were post-cured at 50ºC for two and four hours. Each composite was immersed in the six solutions, except one set that cured at room temperature, which was kept unimmersed as a control for any changes after the specimens were exposed to the corrosive media. Each polymer-mortar composite specimen was weighed before and after immersing. According to the ASTM D2240, shore D Hardness was used to find the hardness of composites by taking six readings on each face of the cylinder. The faces of the cylinder were cleaned from dust and any obstacles to ensure precise readings by the device. The specimen's hardness was calculated by taking the average indentation depth of all readings for each specimen. Specimens were tested using the hydrostatic compression test machine with a maximum capacity of 800KN, according to ASTM C 109 [22], and the maximum compression load for each specimen was recorded. Compressive strength was determined by dividing the force applied to the specimen's surface area by its volume. Figure 2 shows the schematic diagram of the experimental procedure.

Figure 1. Polymer mortar composite specimens.

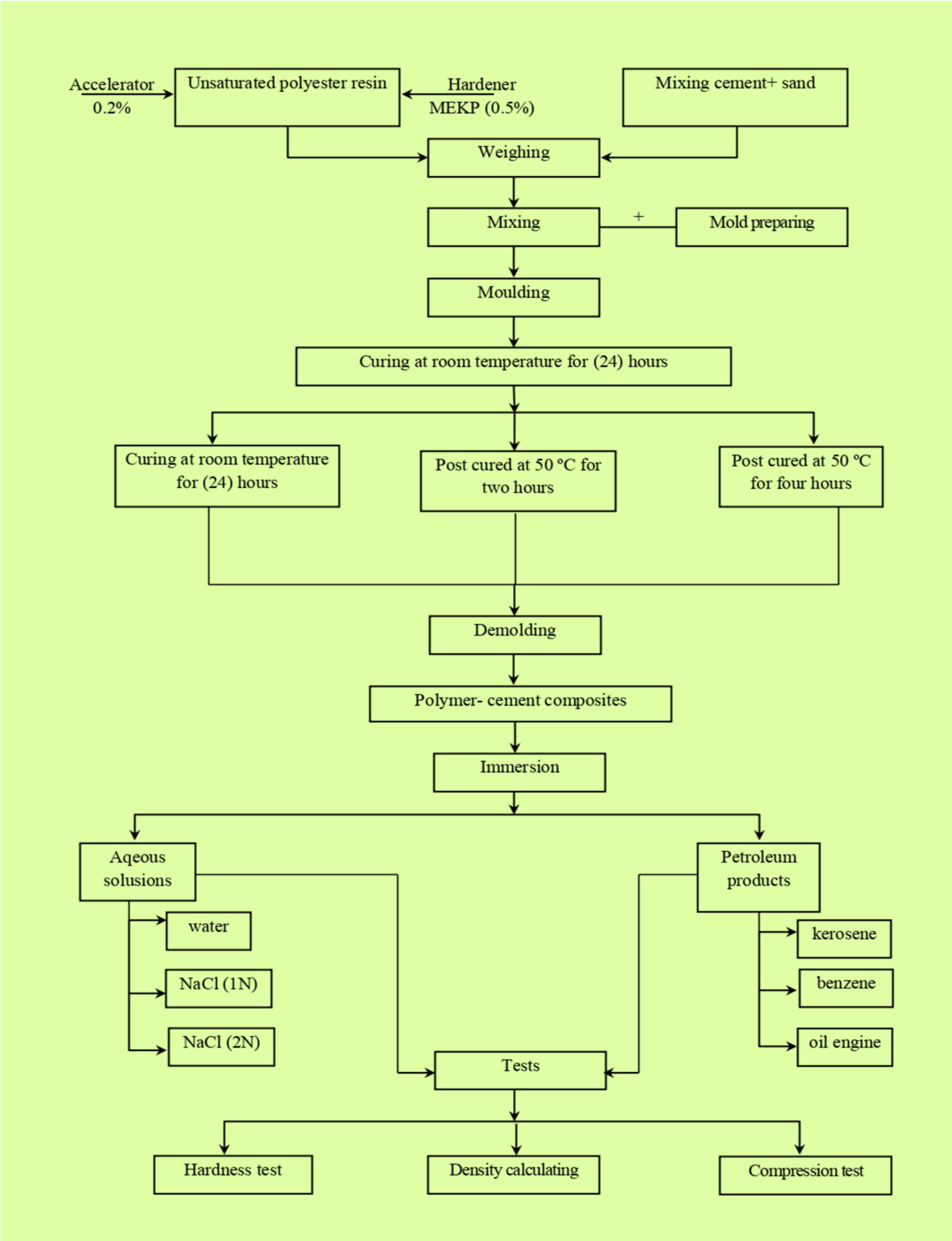

Figure 2. Flow chart represents the basic procedure of the experimental work

3. RESULTS AND DISCUSSION

The building materials are designed for their ability to achieve desired results and their mechanical qualities and general durability. Because of their superior qualities over traditional mortars, polymer-modified mortars have become popular building materials. Polymers have been utilized in mortars and concrete to improve mechanical capabilities, adhesion, and waterproofing characteristics [10, 23].

Because the behavior of polymer-modified mortar might change depending on the test conditions, extra attention to every aspect was necessary. When looking at the behavior of materials in corrosive media, the most common medium to which they are exposed is water. Water may impair the durability of cementitious constructions [24]. When water infiltrates building materials, the hydrostatic pressure rises, causing the construction materials' volume stability and durability to deteriorate [25].

The hardness and compressive strength of polymer modified mortar show that the specimens cured for two hours seem more resistant than other specimens, while the specimens cured at room temperature and post cured for four hours had a behavioral decline. The hardness of the specimen cured for four hours seems to have less hardness than the specimen cured at room temperature; in contrast to the compressive strength which shows the four hours cured specimen is better than that cured at room temperature, as illustrated in Figures 3 and 4. The water absorption clarifies this, as in Figure 5. A polymeric compound may be added to a cementitious material to enhance the interfacial interaction between the matrix and reinforcing material. Increased tensile strength and durability of the material as a result[26-32]. The water causes polymer swelling after a while, and curing the polymer for a suitable period will prevent water absorption and swelling. The curing for two hours is the less absorbing while the highest absorbtion is when cured for four hours which can be explained as an inappropriate curing time that causes degradation in polymer causing micro cracks or may be porosities leading to absorbing water.

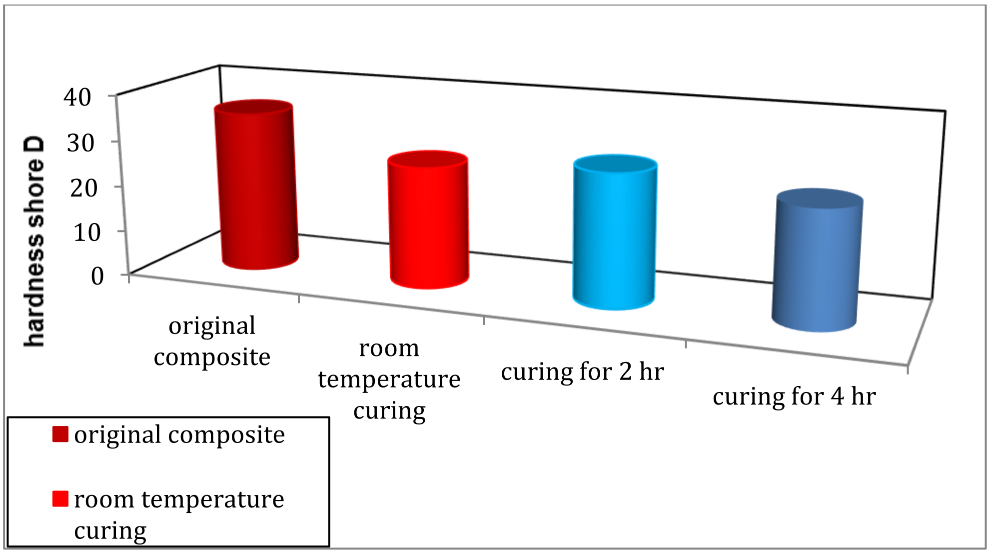

Figure 3. Tap water effect on polymer mortar composite hardness.

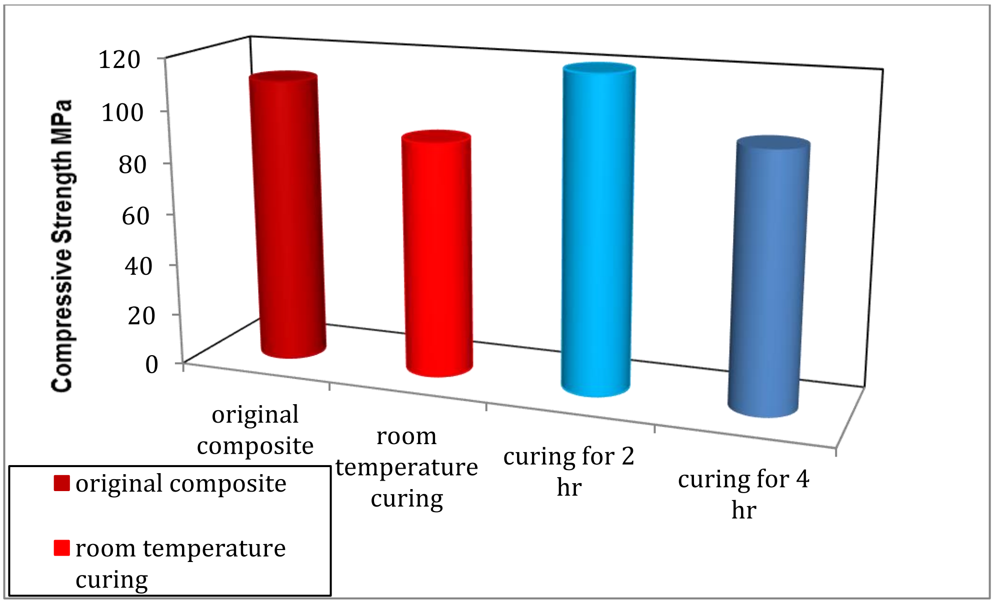

Figure 4. Tap water effect on polymer mortar composite compressive strength.

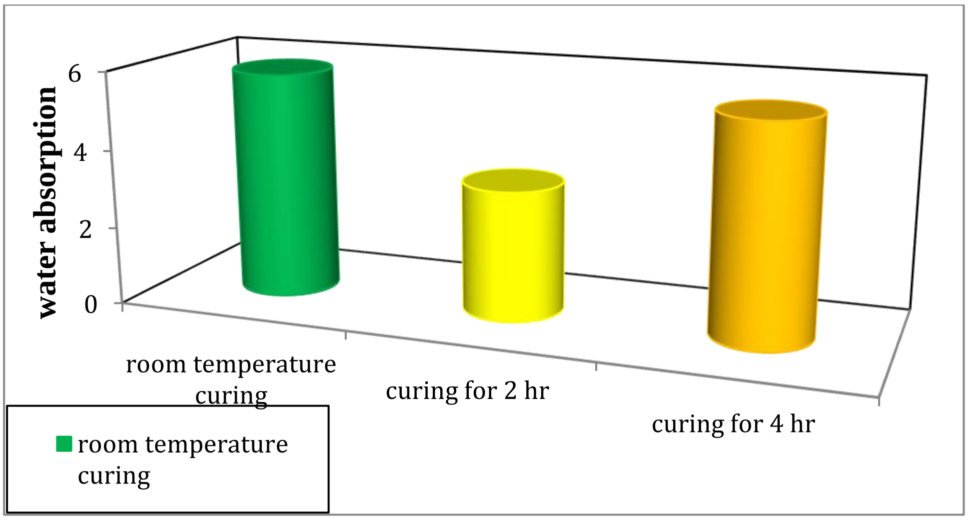

Figure 5. Water absorption of polymer mortar composites.

The behavior of specimens in water can explain the behavior in other media since any liquids can penetrate in the same manner. In every media, the material's surface is the first who encounter any corrosive solution. The salts can cause severe damage to polymer or any construction materials in a short period because salts affect the bonding inside the material, resulting in surface collapse. Figure 6 demonstrates the hardness behavior in 1N and 2N NaCl solutions. It shows that curing at room temperature is more resistant to salts in any concentration. At the same time, the post-curing specimens' uneven behavior according to the concentration of salts i.e. at 1N the two hours is the more resistance but as the concentration increases the post curing at four hours becomes better than the two hours which emphasizes on selecting the adequate curing period. As in water, at any concentration of NaCl, the two hours post-curing is the best compressive strength after immersed in salt solutions, unlike curing for four hours will depend on the salt concentration i.e. at 1N the four hours post curing is better than curing at room temperature on the contrary at 2N, as in Figure 7. According to Viklund et al.[33], the increased resin mobility at higher temperatures might decrease the size and volume of pores, which in turn leads to a drop in compressive strength. Relatively limited attention in the published study was paid to the correct curing of polymer-cement concrete[34].

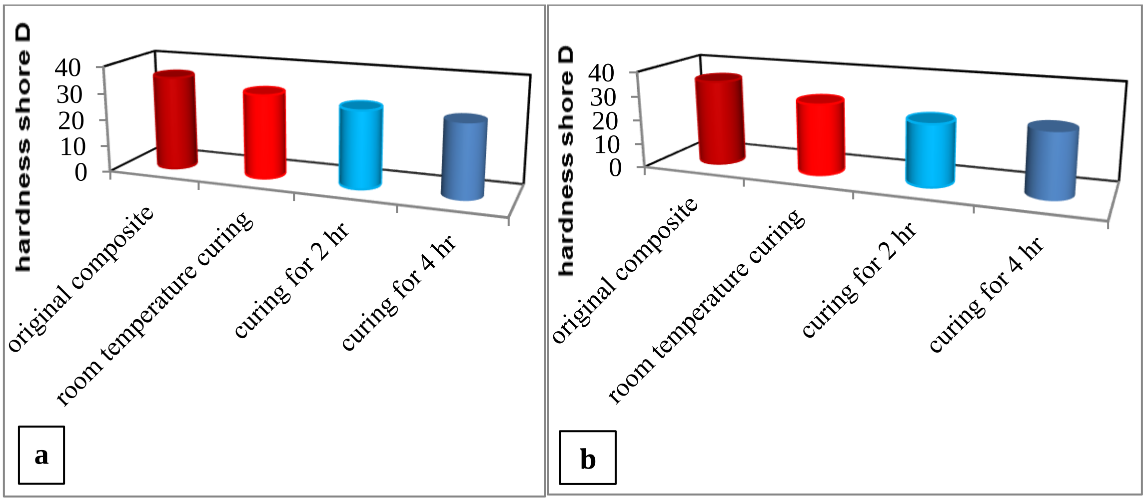

Figure 6. Effect on polymer mortar composites hardness in a) 1N and b) 2N NaCl.

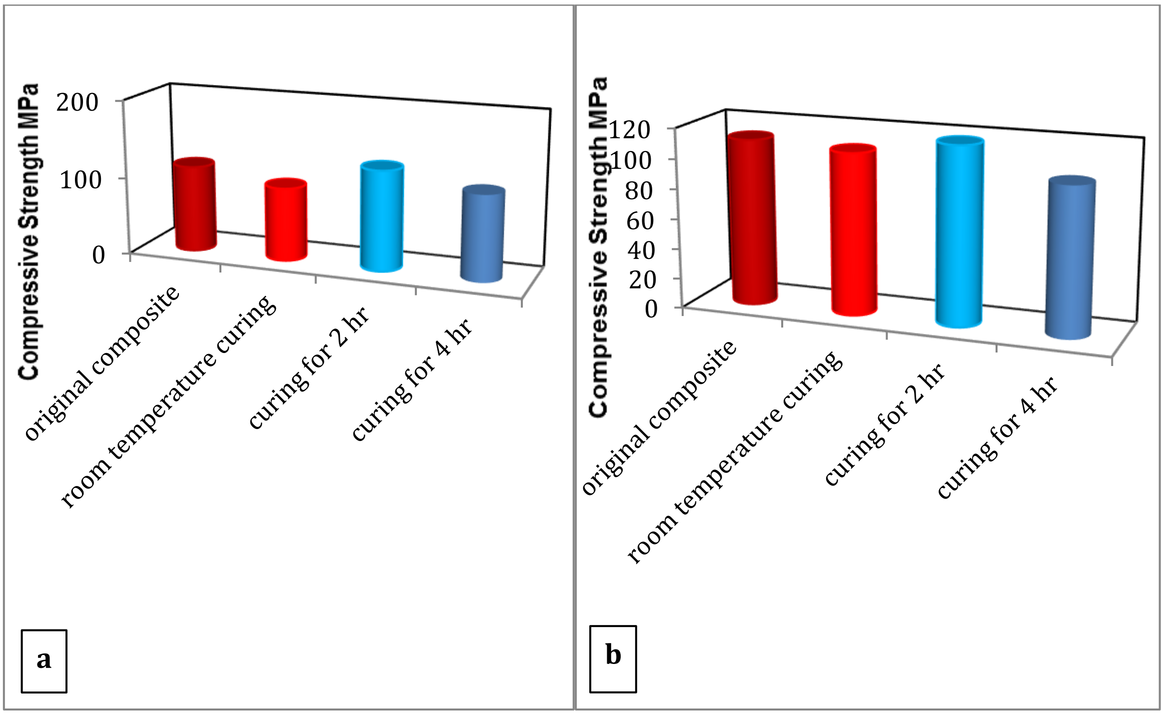

Figure 7. NaCl effect on polymer mortar composites compressive strength in a) 1N and b) 2N NaCl.

Any liquid will search for voids to penetrate the material. It is well known that ceramics are considered porous materials, and construction materials are ceramics. Pores and microcracks provide a path for transporting corrosive substances in construction materials [35-38]. Adding polymer to the construction materials minimizes the void by sealing it [23]. According to the type of oil product, the oil products will try to break the chains in polymers and the interface between the polymer and mortar. In studying hardness after immersion in three different oil products, post-curing will increase resistance to oil products, compact the structure, strengthen the bonding effect between the matrix and the filler, and prevent stress concentration, all of which will help to stabilize the matrix-filler interaction. Except in the case of kerosene, which shows curing at room temperature is more resistant, as illustrated in Figures 8, 9, and 10. Modified mortars were created with a greater understanding of material behavior, particularly in admixtures, and a better understanding of curing processes [39].

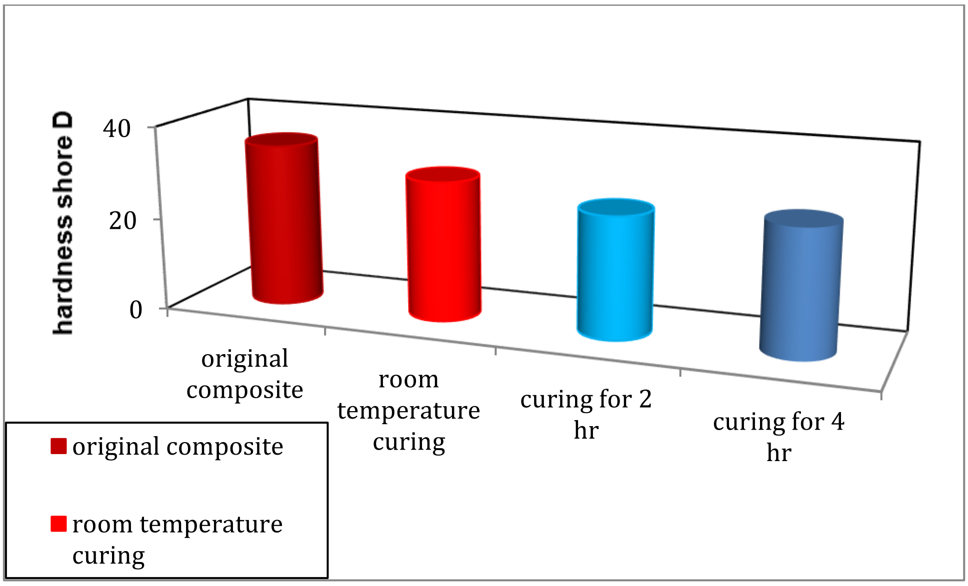

Figure 8. Kerosene effect on polymer mortar composite hardness.

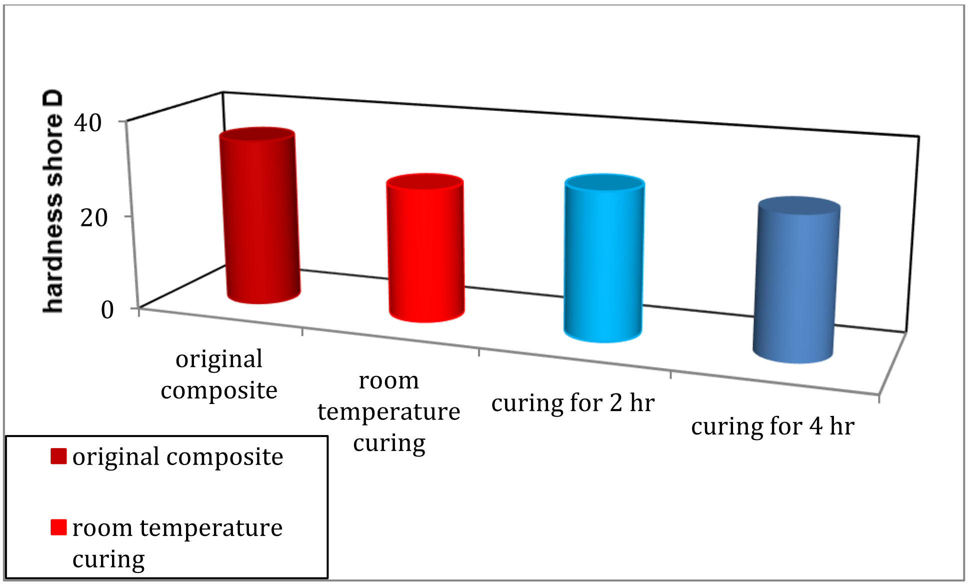

Figure 9. Benzene effect on polymer mortar composite hardness.

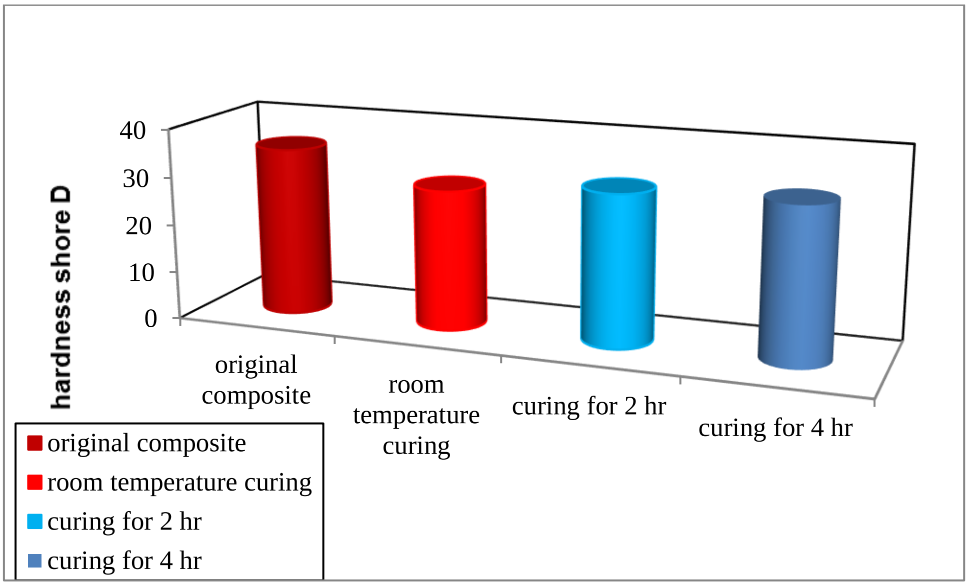

Figure 10. Engine oil effect on polymer mortar composite hardness.

According to the literature, the polymer content or polymer cement ratio has a considerable impact on polymer-modified mortar and concrete characteristics. It refers to the mass ratio of polymer solids in a polymer-based admixture to cement in a polymer-modified mortar or concrete [40-42]. It is well-known in particulate composites that matrix and particles share the compressive strength depending on the particle size and percentage of constituents. The reinforcing in this composite is ceramic, which is characterized as stiffer and harder than the polymeric matrix. Each particle will restrict the movement of the matrix against the applied load. On the other hand, the ceramic structure had higher porosity than other materials. Adding polymer and post-curing will increase bonding between the matrix and reinforce by increasing cross-linking in the structure, giving more resistance to failure. In Figures (11, 12, and 13), the two hours post-curing gave the best compressive strength, whatever the oil product immerse in. Two hours post-curing show the best compressive strength, i.e., more resistance against every corrosive media, as illustrated in Figure 14.

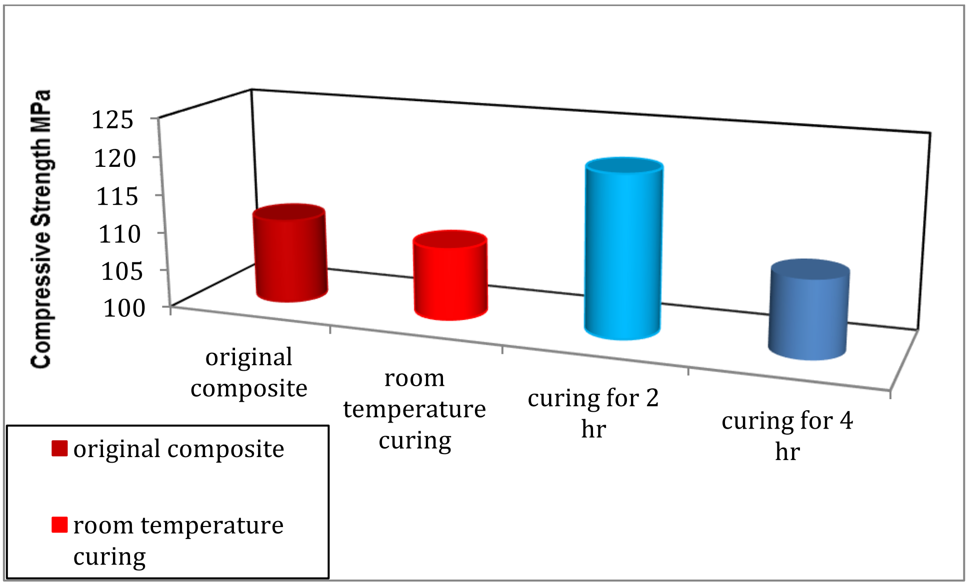

Figure 11. Kerosene effect on polymer mortar composite compressive strength

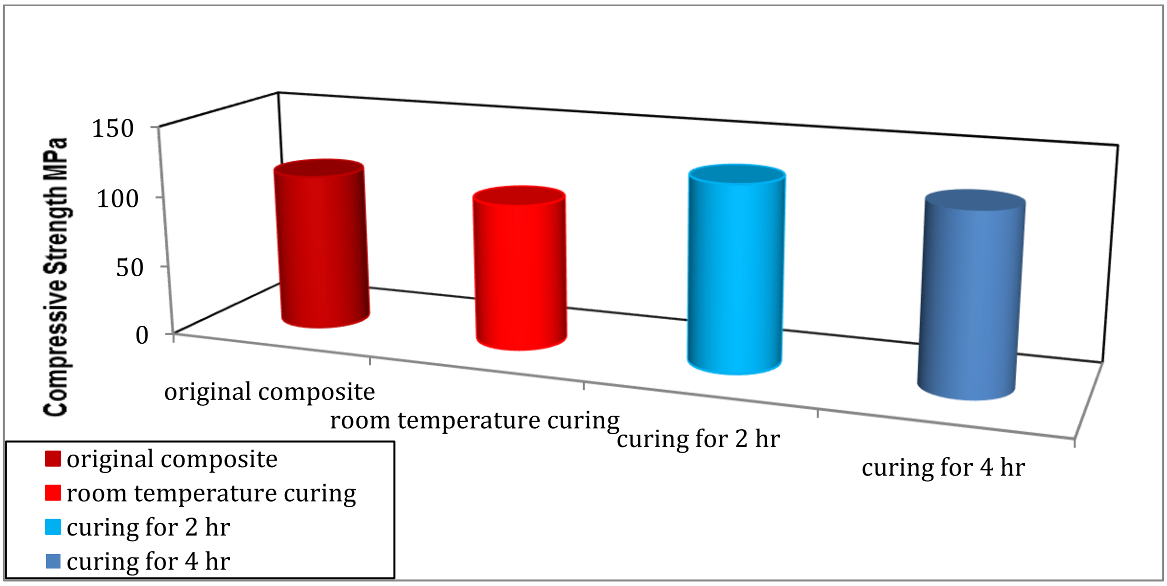

Figure 12. Benzene effects on polymer mortar composite compressive strength.

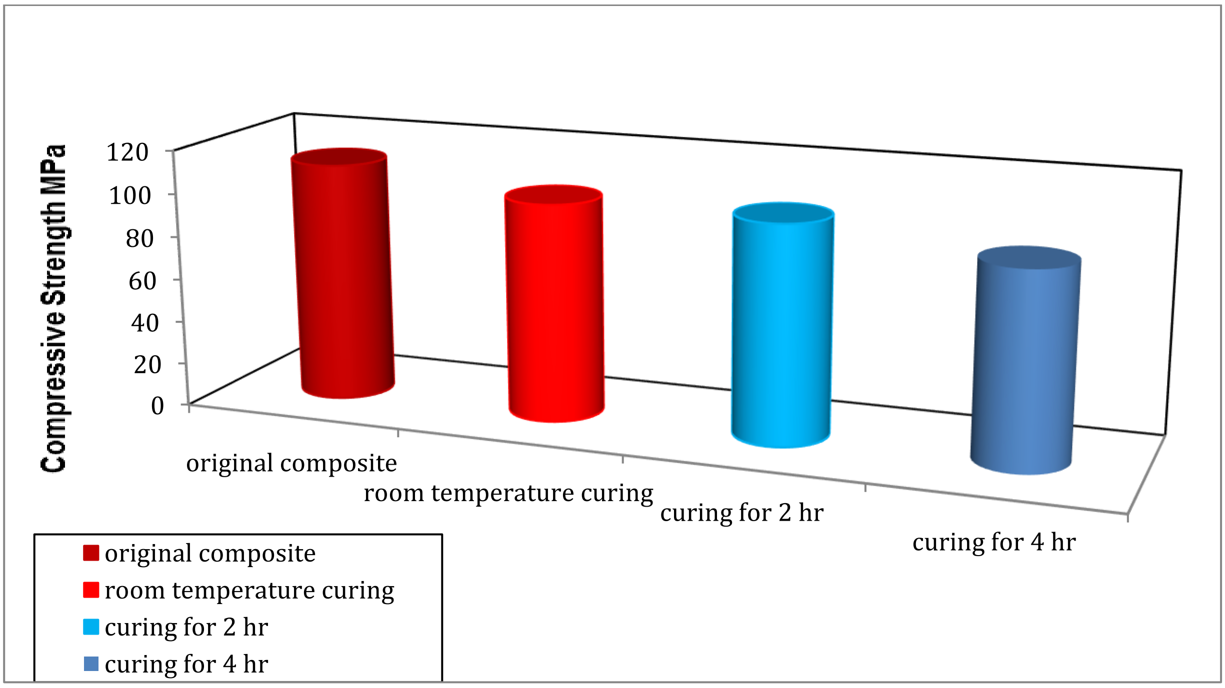

Figure 13. Engine oil effect on polymer mortar composite compressive strength.

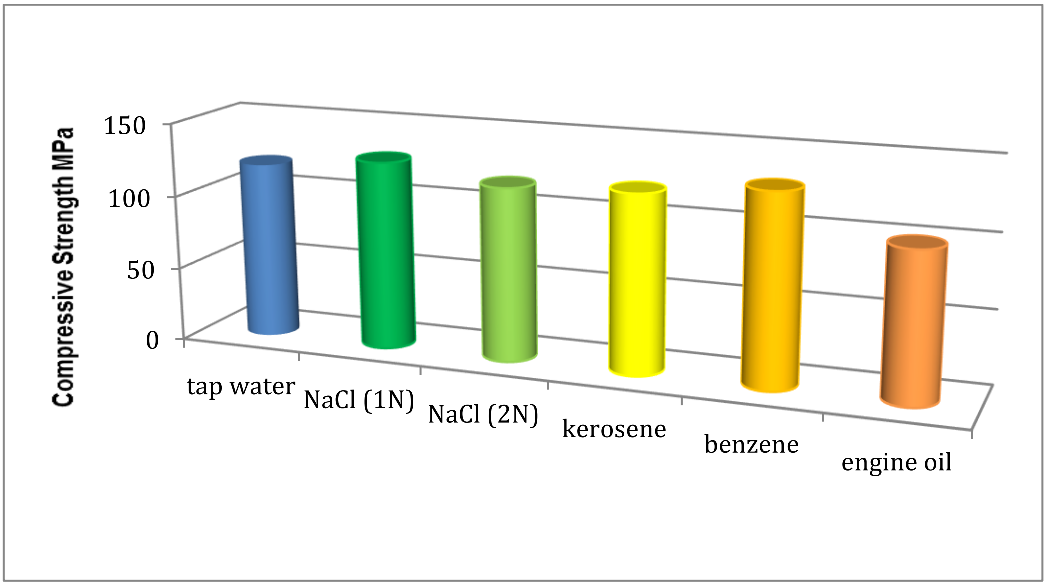

Figure 14. The compressive strength at two hours of curing.

The corrosion rate was determined using the weight changes to assess the impact of corrosive media on the specimens. Tables 1 and 2 demonstrate that the corrosion rate was minimal for all specimens in all media. The specimens submerged in 1N NaCl had the highest corrosion rate. The specimen did not influence after visual examination, except that the edge became rounded after it was sharp, as shown in Figure 15. The water had less effect on the specimens than NaCl solutions. The less effect in the oil products is the engine oil than benzene and kerosene. Hydrophobic impregnation provides an effective physical barrier for building materials, delaying corrosion and lowering internal corrosion rates [43]. Internal humidity, capillary water absorption, and the invasion of water-soluble ions have been considerably reduced [44]. The addition of polymer compounds to building materials improves their hydrophobicity and corrosion resistance [45, 46]. It protects building materials from hazardous chemicals and increases their durability significantly [47-49]. As a result, designing and developing novel polymers to accomplish actual internal hydrophobic alteration has become a significant technology for increasing cement-based products' durability and service life [50].

Table 4 Corrosion rate of polymer- mortar in aqueous solutions.

Solutions curing	Tap water	NaCl (1N)	NaCl (2N)
Room temperature curing	6.77×10^{-4}	9.90×10^{-3}	8.18×10^{-3}
Curing for 2 hr	4.05×10^{-4}	6.89×10^{-3}	6.31×10^{-3}
Curing for 4 hr	7.67×10^{-4}	9.35×10^{-3}	8.922×10^{-3}

Table 5 The corrosion rate of polymer- mortar in oil products.

Solutions curing	Kerosene	Benzene	Engine oil
Room temperature curing	2.54×10^{-4}	7.096×10^{-4}	1.10×10^{-3}
Curing for 2 hr	9.51×10^{-4}	1.03×10^{-3}	1.76×10^{-3}
Curing for 4 hr	2.70×10^{-4}	5.91×10^{-4}	2.025×10^{-3}

Figure 15. Polymer cement samples a) before and b) after exposure to the corrosive media.

4. CONCLUSIONS

Using polymers to modify mortar improves the workability, adhesion, strength, and durability of the cement besides improving cure characteristics. The addition of unsaturated polyester improves cement-sand composites' properties and makes them more resistant to corrosive media. The nature of the media influences the hardness directly. The post-curing can improve both the resistance to corrosive environments and compressive strength, but the crucial issue is the suitable curing period. Exposure to water and NaCl seems to have more impact than oil products. The efficient curing was for two hours more than four hours, which indicates that a longer curing period is not necessarily the best. It is noteworthy in studying the mortar modified by polymer without using water is the absorbtion of any liquid substance had reduced and the behavior of unsaturated polyester can summarized in Table 6:

Table 6 behavior of unsaturated polyester-mortar composites against different solutions

Solutions	**Room curing**	**temperature**	**Post cured for two hours**		**Post cured for four hours**	
	Hardness	Compressive strength	Hardness	Compressive strength	Hardness	Compressive strength
water	decrease	more decrease	increase	increase	more decrease	decrease
NaCl (1N)	more increase	more decrease	increase	increase	decrease	decrease
NaCl (2N)	more increase	decrease	more decrease	increase	decrease	more decrease
kerosene	increase	more decrease	more decrease	increase	decrease	decrease
benzene	more decrease	more decrease	increase	increase	decrease	decrease
engine oil	more decrease	decrease	decrease	increase	increase	more decrease

The table clarify that the polymer with the correct period can utilized to manufacture tanks or pipes that can store petroleum (or oil) products without interaction or corrosion action.

ACKNOWLEDGMENTS

Authors would like to thank Al-Mustansiriya university (www.uomustansiriyah.edu.iq) Baghdad-Iraq for its support in the present study.

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