



A Sustainable Approach for Immobilization Dyeing bath Effluents of Reactive Yellow145 by using Different Types of Eco-Friendly Geopolymer Cement



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Abstract

A suggested approach for handling reactive organic dye waste from the textile industry was developed using the geopolymerization process. Two separate kinds of geopolymer cement pastes are used to reduce the colour of the liquid wastes of reactive yellow 145. According to the results, we were able to cleanse the water created by dyeing processes and lower pollution rates. Geopolymer cement pastes were organized using metakaolin and slag as an aluminosilicate source separately, sodium silicate and sodium hydroxide as an alkali activator in the presence of organic dye pollutant reactive yellow 145. The study was carried out with various PH mediums and hydration times. Spectrophotometric measurements were used to measure the % leaching of pollutant dye. The % leaching of reactive dye was examined with variable pH (0.1M NaOH, deionized water and 0.1M HCl). We also examine the effect of reactive dye on the hydration characteristics of different geopolymer mixes. FTIR and XRD studies were employed to evaluate the influence of the reactive dye's incorporation inside GP samples.

Keywords: Reactive yellow 145 , Slag, Metakaolin, Geopolymer cement.

1. Introduction

There is no doubt that liquid waste was and still is a major factor causing pollution to the environment around us. Efforts to remove pollutants from the natural environment are unable to keep pace. Increasing the amount of waste as a result, our ecosystem is affected by the discharge of liquid pollutants and leads to many health risks. Discharge of color in water is a major concern because it affects living organisms and causes many diseases. There are various methods for removing dyes from sewage discharge such as electrochemical process, coagulation, chemical oxidation, and membrane separation process. Many of these processes are not very popular due to their economic disadvantages and inefficiencies. Among these techniques in our previous research, adsorption was preferred over other techniques for removing dyes from aqueous solutions because of the simplicity of the method and ease of use by using an environmentally friendly geopolymer [1-3]. Geopolymers have gained more

attention during the past twenty years owing to their ability to be used as a high-performance, ecologically friendly building material [4]. In the 1970s, Joseph Davidovits suggested the idea of geopolymer [5]. As opposed to Portland cement-based building materials, geopolymers don't need high-temperature processing and don't produce CO₂, so they may act as a replacement that is both energy- and environmentally friendly. It has also been observed that the engineering characteristics of geopolymeric mortars and concrete match up with those of OPC. Recent research has demonstrated that the permeability of the geopolymeric binder phase is significantly lower than that of Portland cements. This is definitely going to be very helpful in the development of geopolymer technology for areas like coatings to protect transportation infrastructure and tunnels for heavy metal or nuclear waste storage [4]. Geopolymers are three-dimensional inorganic polymeric materials consisting of cross-linked [AlO₄] and [SiO₄] tetrahedral units and alkali metal cations [5]. Geopolymers are often amorphous or partially

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crystalline, and the strength is acquired from the polymerization of -Si-O- and -Al-O- units. By sharing oxygen atoms, $[AlO_4]$ and $[SiO_4]$ tetrahedral units crosslink in geopolymer, and cations such as Li^+ , Na^+ , K^+ , and/or Cs^+ can spread in the pores of the geopolymer network, balancing the negatively charged $[AlO_4]$ units [6,7]. Depending on the raw resources, Metakaolin, quartz, fly ash, and slag are just a few of the natural and industrial waste products that can be used to manufacture geopolymer. A procedure known as geopolymerization reaction, which is regarded as an environmentally beneficial reaction, can be used to create geopolymer, an inorganic polymer. It includes dissolving aluminium silicate with a geological substance or industrial waste, such as slag, ash, biomass, or red mud, and releasing Si and Al species in a very alkaline medium [8]. Metakaolin powders are often used to investigate the mechanisms by which geopolymerization occurs because of their relatively pure composition, which reduces pollution from impurities that are usually present in industrial by-products. Additionally, metakaolin-based geopolymer exhibited finer microstructure, superior mechanical, and superior thermal characteristics [7]. As a result of current development, the water quality is seriously degrading. Contamination with toxic materials such as heavy metals and dyes, which are extremely harmful to both the environment and people, is one of the primary causes. Because it can be made from waste and byproducts, geopolymer adsorbent can be viewed as a sustainable material [9]. Geopolymers (GP) have the potential to be used as adsorbent materials because of their quick strength, ion exchange efficiency, and ability to solidify toxic waste [10]. Toxic compounds can be eliminated from home and commercial effluents by using GP as adsorbents, which has proven to be a viable idea. Modern data has shown geopolymers have the potential to be exploited as effective adsorbents for the removal of heavy metals and dyes from aqueous solutions [2, 3, 11–18]. The removal of hazardous materials via solidification/stabilization techniques is another use for geopolymers [19–22]. This approach benefits the environment as well as requirements and technological best practices [19]. These hazardous wastes are typically mixed with MK, FA, or BFS, then with an alkali activator to solidify them in the GP matrix, which can subsequently be cured to produce solid GP. In comparison to other available technologies, it has numerous benefits, including efficiency of immobilization, economics, or the ability to apply to various types of postprocessing waste. Over the past ten years, there has been an

increasing amount of research that concentrates on the effective use of geopolymer to immobilize heavy metals [19–26]. The elimination of dye-contaminated water as a whole is done with the stabilization/solidification process according to Al-Mashaqbeh *et al* [27]. The solidification technique for dye removal in liquid waste by geopolymerization has received little attention, hence we focus on it in this paper. The purpose of this study is to look into a new, effective technique for solidification and removing reactive organic dye pollutants with the use of cementation materials or geopolymer binders. Additionally, investigate the impact of reactive yellow-145 dye on the hydration properties of different geopolymer pastes. The solidification/stabilization of organic dye pollutant reactive yellow 145 was investigated using two separate geopolymer pastes SF1 [80% slag + 10% fly ash] and MC1 [80% metakaolin + 20% calcium hydroxide]. Our results indicate that the two geopolymers mixes have a good immobilization efficiency for reactive dyes in a range of PH-mediums and hydration time. The leaching % of dye in different leachate solutions according to our study reach to 0 %. As a result, this approach is useful for removing industry waste from dyes.

2. Materials and Experimental Techniques

2.1 Dyeing Procedures

2.1.1 Dyeing of cotton fabric

The reactive yellow 145 dye (0.8 gm 2% shade), was applied with 60 g/L sodium sulphate followed by add 20 g/L sodium carbonate. The dyeing of 40 gm of cotton fabrics was conducted with sodium sulphate at 40 °C for 30 min and then allowing fixation in sodium carbonate for a further 60 min at 60 °C. After a thorough rinsing, the dyed sample was dried at room temperature. The structures of this dyes is shown Figure (1).

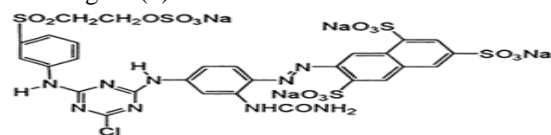


Figure 1. C.I. Reactive Yellow 145

2.1.2 Dyeing of wool fabrics

In a hydrolyzed reactive dye bath containing the remaining different amounts of dye with liquor ratio 50:1, wool fabric was dyed using conventional heating at temperatures 80 °C for 60 min. The dyed samples were rinsed with cold water and dried at ambient temperature. We used the residual dyeing bath effluent in dyeing wool fabric for the second time as a method for treating dyeing baths effluents. It is worth noting that the dyeing operations of cotton or

wool fabrics have taken 27% of the dye concentration, and the remaining dye percentage was 73%, which is equivalent to 0.56 gm/L.

2.2 Materials:

Ground granulated blast furnace slag (GGBFS) is obtained from the Egyptian iron & steel of Helwan Company. Slag had a Blaine surface area of 4700 ± 50 cm^2/g . Its chemical oxide composition is given in Table (1). The XRD pattern is also shown in Figure (2). **Metakaolin (MK)** is supplied by Hemts Construction Chemical Company, Cairo, Egypt. Its chemical composition obtained from X-ray fluorescence (XRF) analysis is presented at Table (1). The XRD pattern is also shown in Figure (2). **Class F fly ash (FA)** is supplied from Sika Chemical Company, Burg Al-Arab, Egypt. Its chemical oxide is given in Table (1). The phase composition of fly ash is investigated using XRD technique as shown in Figure (2). The alkaline activator in this study was sodium hydroxide (NaOH) and liquid sodium silicate (Na_2SiO_3). The NaOH flakes are purchased from EL-Goumhouria chemical company, Cairo, Egypt with purity 99%. Commercial liquid sodium silicate (LSS) is purchased from Silica Egypt Company, Burg Al-Arab, Alexandria, Egypt. The chemical composition of liquid sodium silicate is 11.7 wt% Na_2O , 32.8 wt% SiO_2 , and 55.5 wt% H_2O and silica modulus $\text{SiO}_2/\text{Na}_2\text{O}$ equal 2.80.

2.3. Preparation of geopolymer samples

2.3.1 Preparation of Alkaline activator

The liquid sodium silicate solution and 10M NaOH [28] are mixed together with a fixed ratio of 2:1 in the case of metakaolin-based geopolymer and 1:1 in the case of slag-based geopolymer, until a clear gel is obtained. Then added 100 ml from pre-treated reactive yellow 145 dye to the activator solution and stirred them for half hours before used it.

Table (1): Chemical oxide composition of starting

Oxides, %	MK	FA	GGBFS
SiO_2	64.80	63.10	32.86
Al_2O_3	30.10	26.54	7.02
Fe_2O_3	0.55	5.40	1.14
CaO	0.52	2.33	42.56
MgO	----	0.52	11.58
SO_3	0.13	----	2.50
Na_2O	0.10	----	0.29
K_2O	----	0.09	0.15
Cl	----	0.85	----
TiO_2	2.70	----	----
P_2O_5	0.06	----	----
<i>L.O.I</i>	0.73	----	0.93
Total	99.69	98.83	99.03

materials by XRF, mass%.

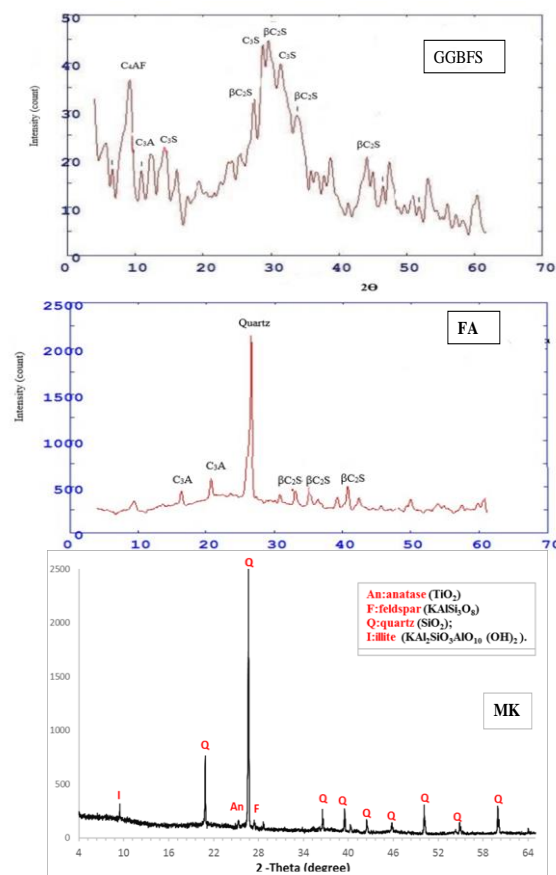


Figure (2):XRD pattern for the raw materials

2.3.2. Geopolymer sample preparation

Different geopolymer pastes are fabricated by mixing dry constituents of each of the following:

1- Mix1 (MC1) contains 80% metakaolin (MK) and 20% calcium hydroxide (CH).

2- Mix 2 (SF1) is made up of 90% Slag (GGBFS) and 10% Fly Ash (FA).

The dry constituents of each mix are handling mixed to attain complete homogeneity. Geopolymer pastes are prepared by mixing raw materials of each mix with the alkaline activator solutions in absence and presence of pre-treated dye, until a homogeneous paste is reached. After complete mixing we confirm the water consistency of the geopolymer pastes by standard Vicat apparatus. Then the pastes are put in stainless steel moulds of one-inch dimension (cubic-shaped molds). The mould is then vibrated for a few minutes to remove the entire air bubbles to give a better compaction of the pastes. The surface of the paste is smoothed by the aid of thin edged trowel. The composition of the various mixes in addition to alkaline activator, dye and the water/solid ratio which gave standard consistency are given in Table (2).

Table (2): Mix composition of the examined mixture and liquid/solid (L/S) ratio.

Mix Name	MK	slag	FA	CH	NaOH ml	Na ₂ SiO ₃ ml	Dye ml	L/S ratio
MC1	80	--		20	117	234	---	0.75
MC1dye	80	--		20	117	234	100	0.75
SF1	--	90	10		69	69	---	0.38
SF1 dye	--	90	10		69	69	100	0.38

2.3.3. Curing:

Immediately after molding, the pastes are cured in relative humidity (100% R.H.) at 60°C for 24 hours. After this period, the cubes are removed from the mould and then MC1 and SF1 cubes cured in H₂O for 3, 7 and 14 days of hydration. While geopolymer mix cubes containing reactive yellow-145 dye are divided into three parts depending on their curing solution type. Specimens are immersed in 100 ml of each of 0.1M HCl, H₂O, and 0.1M NaOH solutions separately to investigate the effect of solution pH on mechanical properties and leaching behavior of different geopolymer-dye mixes at different hydration times.

2.4. Methods of Investigation

1. *The standard water of consistency* are determined according to ASTM C187 [29].

2. *Leaching of reactive yellow-145 dye from geopolymer mixes:* To determine leachability for various hydration times of 3, 7, and 14 days, each geopolymer-dye mix cube contains reactive yellow-145 dye is immersed in 100 mL of the following leaching solutions: 0.10 M NaOH, deionized water, and 0.10 M HCl. After each hydration period, the leachates were filtered and subjected to a UV-Visible spectrophotometer analysis (Spectrophotometer V-670) to determine how much dye was present. So, the cubes were removed from their curing condition and confirmed to the compressive strength and total porosity measurements. Equation 1 was used to measure the leaching percentage (percent L) of organic dye [27].

$$\text{Leaching \%} = (\text{CL}/\text{CT}) \times 100 \quad (1)$$

Where CL is the concentration of the dye leached out (mg/L) of the geopolymer mix cube containing dye and CT is the total concentration (mg/L) of the organic dye incorporated in the geopolymer mix [27].

3. *Compressive strength measurement:* A set of three cubes are used for the determination of compressive strength of all the hardened pastes. The average of the three results is considered expressed in kg/cm². The compressive strength machine used in this work was manually compression testing machine (D550-control type, Milano-Italy)

4. *The stopping of hydration* is performed on the crushed cubic specimens after doing compressive strength test. The resultant crushed specimens were taken, mixed with a stopping solution of alcohol/acetone (1:1) to prevent further hydration, and dried for 24 hours at 50°C before being retained for further analysis (XRD, FTIR).

5. *Total porosity measurement:* Total porosity tests are carried out by determining the weight of samples of hardened pastes suspended in water, W₂, and those in air, W₁ then dried at 100°C for about 24 hours and weight in the air, W₃. The total porosity percentage of (P %) calculated according to the following equation 2:

$$P \% = [(W_1 - W_3) / (W_1 - W_2)] \times 100 \quad (2)$$

6. *pH measurements:* For each geopolymer mix cubes immersed in 100 mL of each of the following leaching solutions: 0.10 M NaOH, deionized water and 0.10 M HCl (in sealed plastic containers) the pH was measured (Adwa AD1000 pH meter) as a function of time (0, 3, 7, 14 days).

7. *The phase characteristics are investigated* by using *X-ray diffraction analysis (XRD)*. The XRD is carried out with a Ni- filtered diffractometer (Empyrean diffractometer, Holland) and Cu-K α radiation of wavelength $\lambda = 1.5418 \text{ \AA}$ and pixel detector operating at the following condition; voltage 40 kV and current 40 mA.

8. *The Fourier Transform Infrared Spectroscopy (FTIR)* measurements are carried out on infrared spectrophotometer (Perkin Elmer-1430 infrared spectrophotometer, USA) using potassium bromide (KBr) pellets. The wave number of infrared spectra was ranging from (400 to 4000 cm⁻¹).

3. Results and discussion

3.1. Effect of reactive yellow-145 dye on physicochemical properties of different geopolymer mixes

3.1.1. Compressive strength

The compressive strength values of hardened alkali activated mixes SF1 and MC1 in the presence and absence of dye cured in different pH media, for different hydration times of 3, 7 and 14 days, are given in Table (3). As we notice from the table, the values of the compressive strength of all SF1 geopolymer mixes (with and without dye) increase with curing age, except in the case of HCl medium, which decreases with time. The values for compressive strength for SF1-dye mix curing in alkali and neutral media increased significantly than SF1 mix, as a result of the dye's incorporation into the slag based geopolymer matrix. On the other hand, the compressive strength values of MC1 and MC1-dye mixes dropped with hydration time and were negatively influenced by the dye's incorporation into

the geopolymer matrix at all investigated media. Additionally, we noted from Table (3) that the compressive strength values for cubes immersed in an acidic solution are significantly close to the values of cubes immersed in other media, unlike what was expected to decrease as a result of acid attack. This indicates the acidic resistance of geopolymer pastes and is attributed to the formation of NaCl as a result of the chemical reaction between NaOH and HCl. According to a prior investigation [34], NaOH reduces the impact of the HCl attack.

Table (3): Compressive strength (kg/cm²) of SF1 and MC1 at different hydration ages

Geopolymer mix	Compressive strength (kg/cm ²)		
	3 days	7 days	14 days
SF1 without dye	360	375	420
SF1-dye curing in H ₂ O	350	380	450
SF1-dye curing in 0.1M NaOH	345	400	430
SF1-dye curing in 0.1M HCl	340	330	320
MC1 without dye	300	260	210
MC1-dye curing in H ₂ O	200	160	130
MC1-dye curing in NaOH	200	170	120
MC1-dye curing in HCl	180	140	100

3.1.2. Total porosity (P%)

The total porosity of hardened SF1, SF1-dye, MC1 and MC1-dye specimens cured for 3, 7 and 14 days are seen in Table (4). For all mixes, it has been demonstrated that total porosity decreases with curing time. This is explained by the pozzolanic activity of metakaolin and slag, which react with calcium hydroxide and fly ash, respectively, and produce extra amounts of hydration products. These hydration products precipitated in certain open pores that were available, reducing the total porosity. The total porosity values under various pH's media and up to 14 days of curing are slightly influenced by the addition of reactive yellow dye in the geopolymer matrix.

3.1.3. X-ray diffraction analysis (XRD)

XRD patterns of alkali activated mixes SF1, SF1-dye, MC1 and MC1-dye after 3 days of hydration curing in water are shown in Figure (3). The figure shows a significant quartz peak that appears at $d=3.34\text{\AA}$, the unreacted silica from the raw materials may be the cause of this peak. For SF1 and SF1-dye geopolymer mix, XRD diagrams show broad and diffuse peaks around $d = 2.78\text{--}3.34\text{ \AA}$, implying amorphous or short-ordering structure phases generally present in alkali-activated GBFS-FA. The significant peak at d

$= 3.05\text{\AA}$ in alkali activated GBFS-FA is attributable to the presence of calcium silicate hydrate phase [30, 31]. Quartz and illite impurities are present in the geopolymer pastes of MK-CH systems, and the intensity of the quartz peak in these systems is larger than in the Slag-FA mix systems. Additionally, we can see that the C-S-H phases are responsible for the amorphous to semi-crystalline phases that are seen in the XRD patterns of the MC1 and MC1-dye mix systems at (3.05\AA) . The type and intensity of the hydration product produced are unaffected by the presence of the dye in geopolymer pastes, as seen in Figure (3).

3.1.4. Fourier Transform infrared spectroscopy (FTIR)

Figures (4) illustrate the FTIR of metakaolin and slag-based geopolymer mixes with and without reactive yellow-145 dye (MC1, MC1-dye, SF1 and SF1-dye) after 3 days of hydration. The stretching vibration mode of the O-H group, which is located at $3442\text{--}3460\text{ cm}^{-1}$, and the bending vibration mode of the H-O-H group, which is at $1644\text{--}1649\text{ cm}^{-1}$, both were associated with absorption bands in all spectra. This suggests that raw material species are dissolved and condensed using the alkalis in additives and the alkaline activator solution, resulting in the creation of an excessive amount of hydration products inside the geopolymer matrix [32, 33]. The band is observed between 1450 and 875 cm^{-1} due to the carbonation process, namely the stretching vibration of the C-O bond in CO_3 groups. The peak in the range $1016\text{--}1000\text{ cm}^{-1}$ related to the asymmetric stretching vibration of Si-O-T (T= Si or Al), for geopolymer mixes. Also, the shift of these band to lower wave number can be interpreted as a result of the geopolymerization process and formation of amorphous aluminosilicate gels (C-S-H and N-(C)-A-S-H) in geopolymer binders. This is consistent with the intensity of the bands in the range $775\text{--}711\text{ cm}^{-1}$ and 693 cm^{-1} that correspond to the symmetric stretching vibration of (Si-O-Si) and (Si-O-Si or Al-O-Si), respectively. The reducing of the intensities of the sharp peak of alumina at 798 cm^{-1} was also an indication for the geopolymerization reaction. Finally, the presence of bending vibration mode of Si-O-Si at about $(449\text{--}451)\text{ cm}^{-1}$ related to unreacted quartz. As we noticed from Figure (4), the presence of reactive yellow-145 dye has a reducing effect on the carbonation process and the amount of unreacted quartz. On the other hand, the shift in the value of the asymmetric stretching vibration of Si-O-T from 1016 to 970 cm^{-1} in MC1 system and from 1007 to 1003 cm^{-1} in SF1 system is another induction of the positive impact of dye on the geopolymerization process

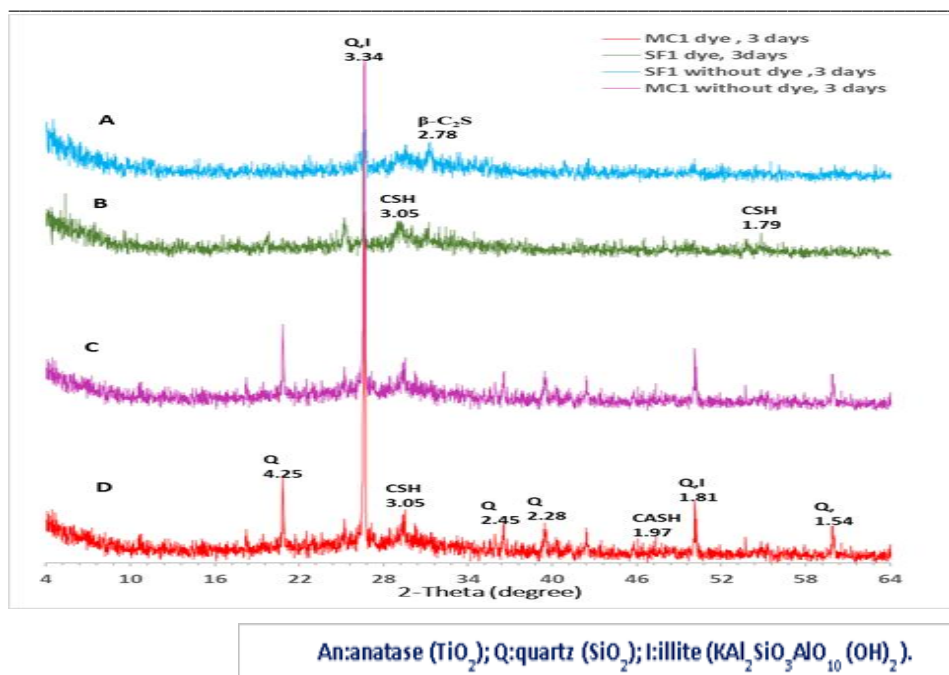


Fig (3): XRD diffractograms of different geopolymer mix cured in water at 3 days ,A) SF1-dye, B) SF1 , C) MC1 , D) MC1-dye

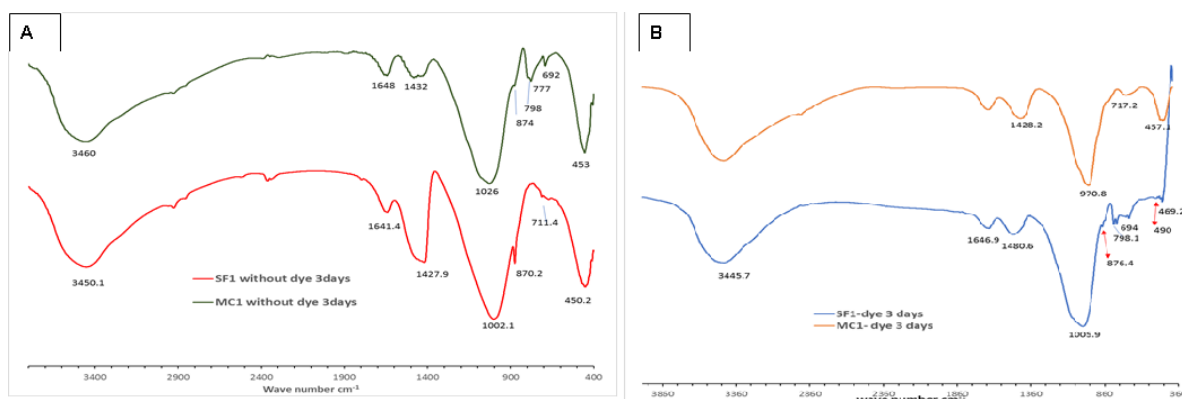


Fig (4): FTIR spectra of different geopolymer mix cured in water after 3 days, A) without dye, B) with reactive yellow-145 dye

Table(4): Total porosity (P %) of SF1 and MC1 at different hydration ages.

MC1	Time			SF1	Time		
	3 days	7 days	14 days		3 days	7 days	14 days
In H ₂ O	31.15	30.56	29.88	In H ₂ O	34.42	33.05	31.85
With dye In H ₂ O	32.25	32.04	31.88	With dye In H ₂ O	33.95	32.95	31.66
With dye In NaOH	32.15	31.89	31.25	With dye In NaOH	33.78	32.25	31.58
With dye In HCl	32.22	31.95	31.45	With dye In HCl	34.66	33.25	32.86

Table (5): pH's values of leachate solutions of SF1-dye and MC1-dye at different hydration ages.

Geopolymer mix	pH				
	0 h	3h	3 days	7 days	14 days
SF1-dye curing in H ₂ O	7.20	12.75	13.23	13.38	13.58
SF1-dye curing in 0.1M NaOH	12.33	12.77	13.33	13.40	13.47
SF1-dye curing in 0.1M HCl	1.76	11.03	13.05	13.16	13.30
MC1-dye curing in H ₂ O	7.20	11.65	12.54	12.65	12.75
MC1-dye curing in 0.1M NaOH	12.33	11.78	12.74	12.86	12.98
MC1-dye curing in 0.1M HCl	1.76	9.88	12.31	12.54	12.56

3.2. Leaching of reactive yellow-145 dye from geopolymers.

3.2.1 Absorbance measurements for leaching solutions as a function of contact time with geopolymer mixes.

The efficiency of immobilization 0.56 gm/L effluents of reactive yellow-145 dye inside the geopolymer matrix was studied using the absorbance measurement of leachate solutions after different hydration time (3, 7 and 14 days). The effect of different leaching solutions pH's (deionized water, 0.1 M NaOH and 0.1 M HCl) on the leachability of the reactive yellow-145 dye from geopolymer mixes cubes is shown in Figure (5 and 6). Figures (5& 6) show also the absorbance band spectra of reactive yellow dye with λ_{max} at 419 nm. Figures 4 and 5 show that the absorbance of different leachate solutions at the λ_{max} of reactive dye (419 nm) for different geopolymer-dye mixes equals zero after 14 days of hydration. This could be because the geopolymer matrix and its hydration products (CH and CASH) have excellent adsorption properties, which contribute in the removal of pollutants such as heavy metals and organic dyes [35].

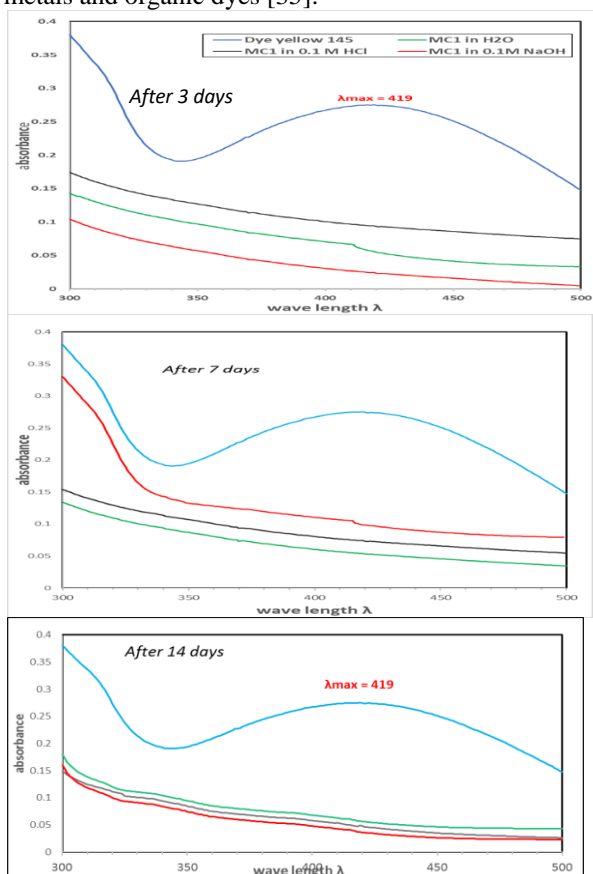


Fig 5. Absorbance band spectra of dye and different leachate solutions for MC1-dye mix at different hydration age

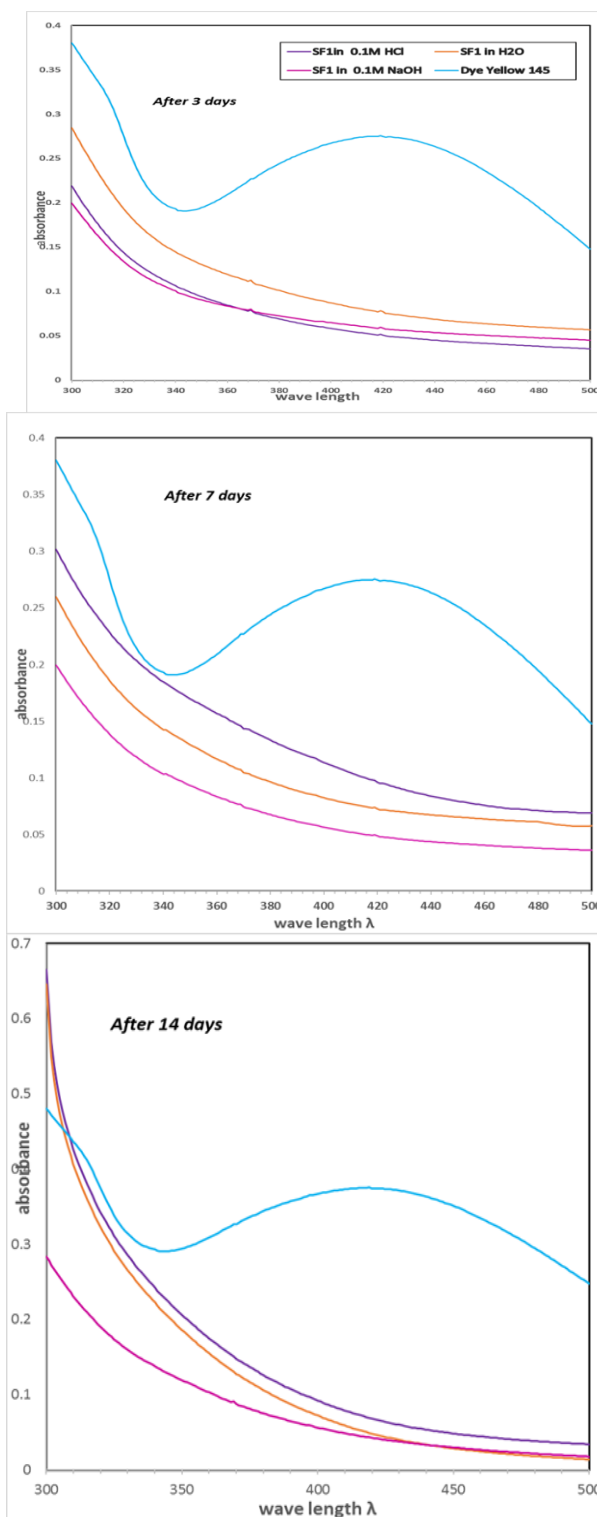


Fig (6): Absorbance band spectra of dye and different leachate solutions for SF1-dye mix at different hydration age

3.2.2. pH measurements for leaching solutions as a function of contact time with geopolymers

For various geopolymer mixes cubes containing reactive yellow-145 dye (SF1-dye & MC1-dye), the pH of leaching solutions was investigated as a function of contact time (0, 3 hours, 3, 7 and 14 days) as shown in table (5). The pH values were increased with time for various types of leachate solutions till 14 days of hydration. These high values are attributed by the geopolymer matrix's progressive hydration over time and the release of alkaline species into leaching solutions [27]. Furthermore, after 3 hours of hydration, the pH values for SF1-dye and MC1-dye, respectively, were 11.03 and 9.88, which are lower than in the case of alkali and neutral medium. This is due to the acidic leaching solutions (0.10 M HCl) neutralized the alkali ions released from the geopolymer mixes.

Conclusion

In this work, two different geopolymer cement pastes based on slag and metakaolin were prepared, and their ability to remove the reactive yellow-145 dye was examined. Our results show that the two eco-friendly geopolymer pastes used have a 100% dye removal efficiency. In addition, the physicochemical properties of several prepared geopolymer pastes in the presence and absence of reactive dye studied examined. The results indicate that dye has a slight effect on the compressive strength and total porosity values of SF1 and MC1 mixes while having no effect on the type of hydration products formed.

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