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Immobilization of Some Heavy Metals in Geopolymer Based on Water Treatment Sludge and Alum Industry Solid Waste

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ABSTRACT

This paper investigates the immobilization behaviors of Cd^{2+}, Pb^{2+} and Hg^{2+} ions in a geopolymer based on water treatment sludge (WTS) and dealuminated metakaolin (DaMK) solid waste from alum industry. For synthesis of WTS/DaMK based geopolymer, five mixes of WTS/DaMK (75/25, 80/20, 85/15, 90/10 and 95/5, respectively) are tested to obtain the optimum synthesis condition based on the compressive strength. Results showed that the geopolymer mortar with 85/15WTS/DaMK has the highest compressive strength (8 MPa). In addition, X-ray Diffraction (XRD) and X-ray Fluorescence (XRF) techniques are used to characterize the mineral and chemical compositions of WTS and DaMK. WTS/DaMK based geopolymer mortar have been tested for leaching to study its immobilization behavior under the optimum condition. The Cd^{2+}, Pb^{2+} and Hg^{2+} ions were used and effectively immobilized in this study. The heavy metals with concentrations of 100, 200 and 300 ppm were used in the geopolymer matrix giving about 98% of immobilization efficiency. The study showed that Pb^{2+} has the best immobilization efficiency followed by Hg^{2+} and Cd^{2+} with high concentrations.

Keywords

Geopolymer, dealuminated metakaolin, water treatment sludge, compressive strength, Heavy metals, immobilization.

1. INTRODUCTION

Geopolymer term has developed greatly in recent years as a new type of cementitious or binder material [1, 2]. Geopolymer is distinguished from ordinary Portland cement by; production of cement requires higher calcining up to 1500°C in the rotary kiln, while geopolymer needs 600–800°C; Portland cement emits CO_2 more 10 times than geopolymer during the calcination; geopolymer resists chemicals and high temperatures giving good strength at room temperature, with excellent solidification of heavy metal ions [3, 4]. This make geopolymer with great properties in fields of civil, military engineering and environment like bridges, pavements, tiles, waste treatment, hydraulic, underground and insulators [5, 6].

Water treatment sludge (WTS) is generated as waste which required in conventional water treatment include disinfectant; clarification (coagulation, flocculation and sedimentation) and sand filtration are used to remove microorganisms, organic matter and suspended solids from surface water. Due to the cost of disposal and the prosecution by the Environmental Protection Agency, reusing water treatment sludge to manufacture ceramics and bricks has been considered as an eco-friendly alternative to landfills or drains [7, 8, 9].

Dealuminated metakaolin (DaMK) is a byproduct of aluminum sulfate industry, the SiO_2/Al_2O_3 ratio and B.E.T values are increased [10, 11]. It is reported that DaMK shows an appreciable pozzolanic activity using isothermal calorimetric tests [12]. The investigation of DaMK confirmed presence of quartz and highly amorphous silica with a high surface area indicating high reactivity [13].

Millions of tons of waste including heavy metals from mine tailings, electroplating, dyeing and more industrial sludge are produced as waste each year worldwide. These wastes are accumulated in the ecosystem with a vast disposal issue. In general, Cd^{2+}, Pb^{2+} and Hg^{2+} are common toxic metals [14]. Therefore, studies on the immobilization these metals are required for testing new solidification materials. Pb^{2+}, Hg^{2+} and Cd^{2+} metal ions were participated in building of the geopolymer structure and can be adsorbed by the aluminium ions on the geopolymer skeleton. Based on this, the heavy metal immobilization is controlled by the adsorption mechanism and physical encapsulation according to the methodology in [15].

Previous studies were focused on geopolymers produced from metakaolin alone [16, 17, 18, 19] or wastes e.g., fly ash and slag [20, 21, 22]. Some studies were focused on using WTS and DaMK as a waste immobilizing agent worldwide [21, 22, 23, 24, 25. 26]. However, WTS and DaMK contain much reactive SiO_2 and Al_2O_3 that are suitable raw materials.

This study aims to produce a geopolymer mortar using WTS/DaMK and investigate its immobilization behaviour incorporating Cd^{2+}, Pb^{2+} and Hg^{2+} ions as an eco-friendly approach for waste management and reducing the waste budget disposed into landfill and drains.

2. MATERIALS AND METHODS

2.1 Materials

WTS samples were collected from Sheikhzaied Water Treatment Plant located in sheikhzaied city, Giza, Egypt. The sludge consists of a ratio of alumina from the coagulation process in the Water Treatment Plant. Calcining temperature for preparing WTS was about 700^{o}C for 1 h. DaMK samples were obtained from the Egyptian Aluminum Sulfate Company (ASCE), Egypt. WTS and DaMK samples were grinded to fine powder, followed by grain size analysis. We used the grain size analysis to identify the grinding status of the studied wastes which is fine or course in order to keep the homogeneity of the mixes and to prevent presence of voids or cracks within the geopolymer mortar. Moreover, the compressive strength of geopolymer mortar relatively decreases with high grain size [27]. The chemical composition was obtained by XRF and the crystalline phases by XRD. The geopolymerization reaction between WTS/DaMK mixtures is occurred when NaOH is adding as a solution alkaline activator. However, the compressive strength is very important and a unique feature in identifying the solidification of geopolymers and cementitious materials [28]. $Cd(NO_3)_2$, $Pb(NO_3)_2$ and $Hg(NO_3)_2$ are used as heavy metal sources.

2.2 Sample preparation

2.2.1 Synthesis of WTS/DAMK based geopolymer

Five various batches of geopolymer are carried out in this study. Batch WTS/DaMK has different weight ratio from 75/25, 80/20, 85/15, 90/10 into 95/5 respectively. Oxides (in molar ratios) are optimized as SiO_2/Al_2O_3, Na_2O/Al_2O_3 and H_2O/Na_2O, therefore batch ratios will be selected in the range that achieves around SiO_2/Al_2O_3 = 4.5 to comply with previous studies in comparison to the influence on mechanical strength [29].

The WTS/DaMK batches are dry mixed for 5 minutes then water was added during mixing and NaOH is mixed for another 5 minutes and cooled to room temperature. Sand is added to WTS/DaMK and mixed together. The produced geopolymer is moulded in (50mm×50mm×50mm) cubes for testing the compressive strength and vibrated for a half minute. A plastic film covers the top surface of moulds to eliminate the water evaporation. Samples are excavated and cured under room temperature after one day.

2.2.2 Efficiency of WTS/DAMK immobilization

Three heavy metal ions Cd^{2+}, Pb^{2+} and Hg^{2+} are mixed with WTS/DaMK geopolymer as a solution of $Cd(NO_3)_2$, $Pb(NO_3)_2$ and $Hg(NO_3)_2$ in water with concentrations of 100, 200 and 300 ppm. Batch “WTS/DaMK-Cd100”, “WTS/DaMK-Cd200” and “WTS/DaMK-Cd300” are utilized to study the immobilization efficiency of Cd^{2+} concentration, Batch “WTS/DaMK-Pb100”, “WTS/DaMK-Pb200” and “WTS/DaMK-Pb300” are utilized to study the immobilization efficiency of Pb^{2+} concentration, and Batch “WTS/DaMK-Hg100”, “WTS/DaMK-Hg200” and “WTS/DaMK-Hg300” are utilized to study the immobilization efficiency of Hg^{2+} concentration.

2.3 Experiment and tests

2.3.1 Mechanical tests

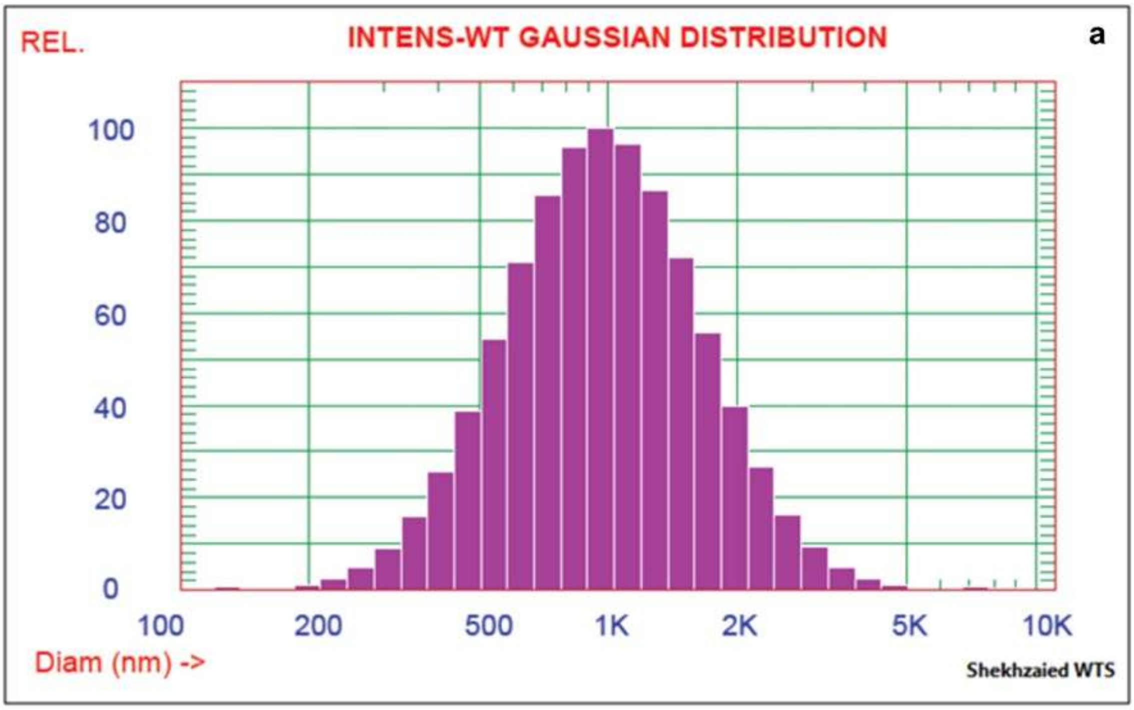
Compressive strengths of the WTS/DaMK geopolymer cubes were tested after 7 and 28 days of curing according to the ASTM C109 Method [30, 31]. The compressive test was carried out using Non-Automatic Compression Range 200 KN-Hoek Cell Machine in the Material Test Laboratory at National Research Centre (NRC), Cairo, Egypt.

2.3.2 Leaching tests

Leaching test using Toxicity Characteristic Leaching Procedure (TCLP) is carried out in this study [21, 32, 33]. The TCLP test is carried out according to [21]. We extracted samples at one, two, three, four, twenty and twenty-four hours. These samples were analysed for heavy metals using ICP-AES in the Reference Laboratory of Drinking Water at the Egyptian Holding Co. Water and Wastewater (HCWW), Cairo, Egypt.

3. RESULTS AND DISCUSSION

The particle size distribution of WTS and DaMK samples is shown in Fig. 1, indicating that WTS particles are irregular in shape within a range of 680–1511.7 nm with a mean of 1115.3 nm. DaMK particles are slightly finer and spherical within a range of 188–514 nm with a mean of 366 nm. The analysis shows that 25% from WTS particles are closely like 80% of DaMK [34].



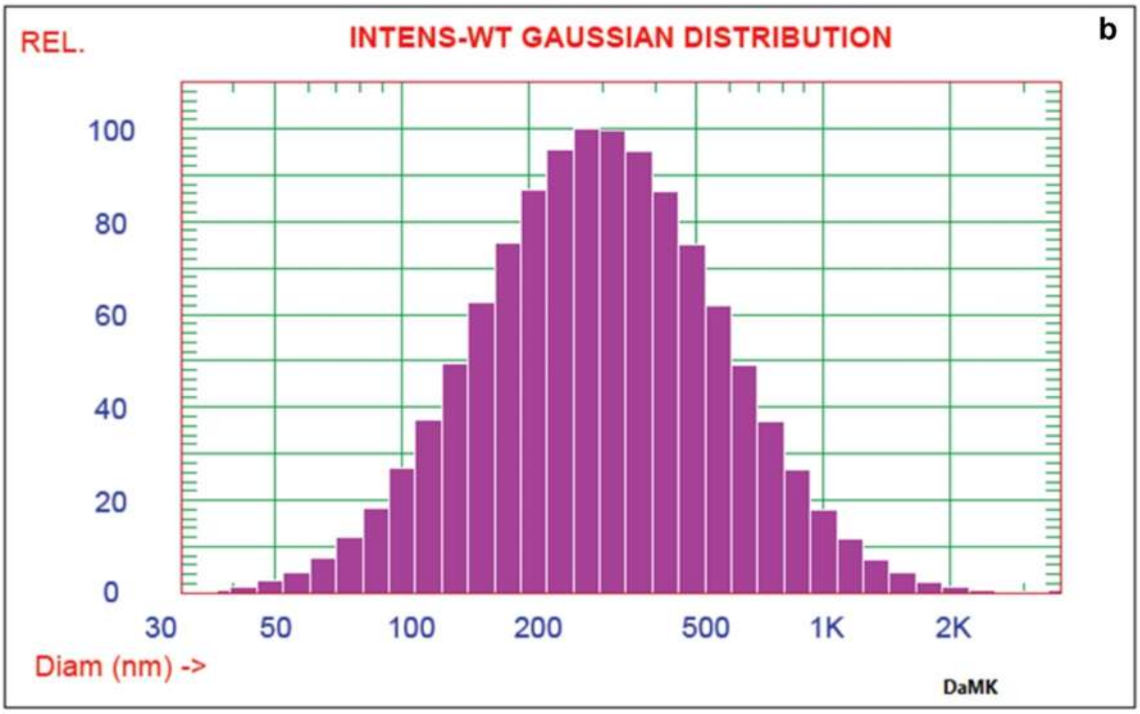


Figure (1): Particle size distribution of WTS (a) and DAMK (b).

The value of specific gravity of aggregate and filler is very important in determining the quantity, which required in the mix proportioning. The specific gravity of sand, which used in this study, is 2.35 and carried out using laboratory tests [35, 36].

The WTS sludge consists of a ratio of alumina from the coagulation process in the Water Treatment Plant, and then it was calcined at 700^{o}C for 1 h in mafle furnace at HCWW. While, kaolinite is the raw material in the manufacture of MK and DaMK after several successive operations that are carrying out in the ASCE, where kaolinite is cracked, grinded and then calcined at 700°C for two hours to form MK, then react with concentrated sulfuric acid to extract alumina and forming DaMK [37]. The mineral characteristics of WTS and DaMK samples are determined by XRD and shown in Fig. 2. The analysis shows that WTS samples consist of mainly halo humps with peaks of minor crystalline inclusions of hatrurite, chloromagnesite and quartz (Fig. 2a). While DaMK samples consist of amorphous humps with peaks of mineral components of quartz, anatase and sillimanite (Fig. 2b).

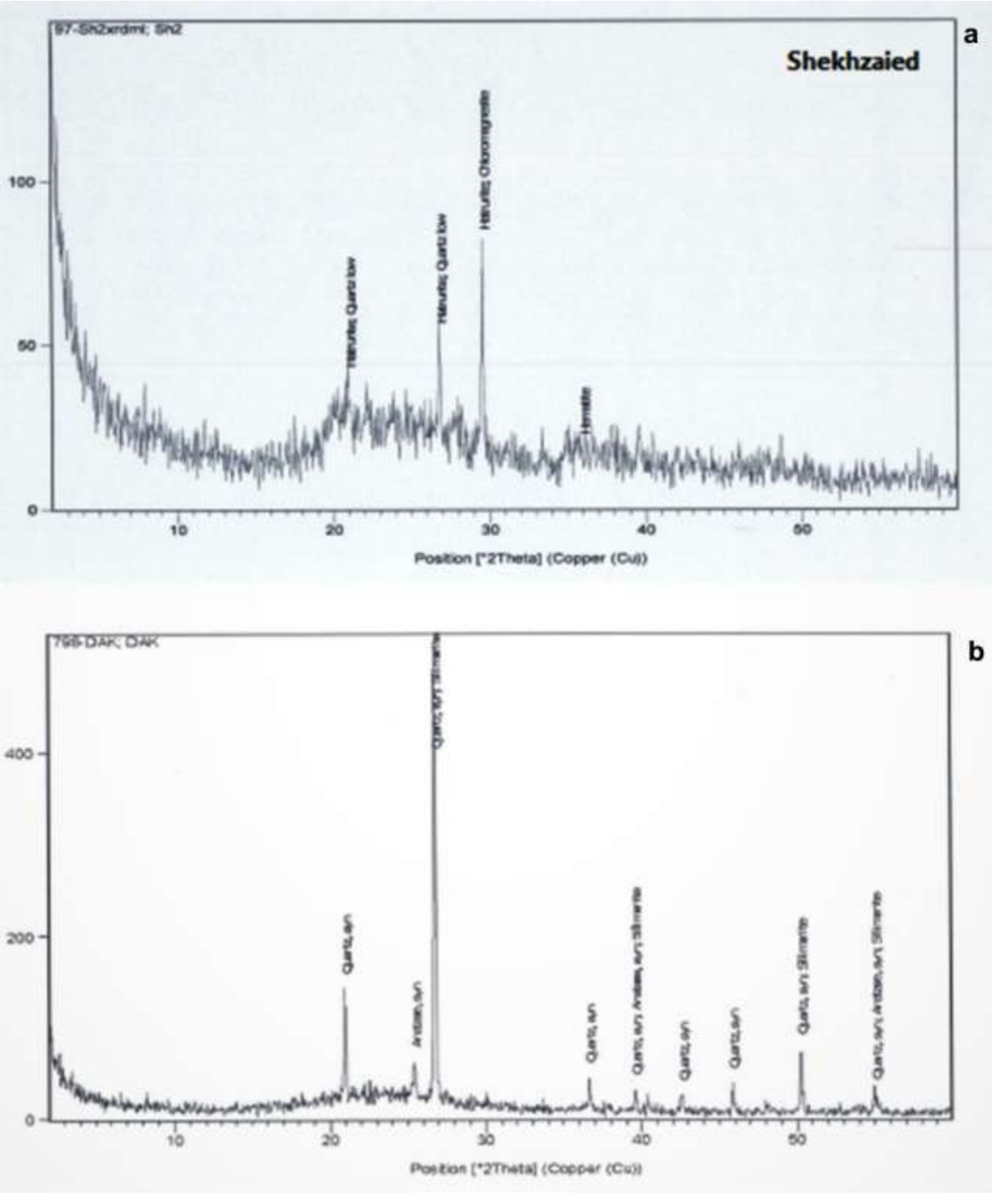


Figure (2): XRD patterns of WTS (a) and DaMK (b).

Table 1 shows the chemical criteria of WTS (dried at 105^{o}C and calcined at 700^{o}C) and DaMK samples which determined by XRF analysis to check whether the materials are a pozzolan and if it can be used in production of geopolymer mortar. The analysis shows the percentages of major components (SiO_2, Al_2O_3 and Fe_2O_3) in WTS105 are 58.36% while CaO and SO_3 contents are 5.46% and 1.92%. After calcinations at 700°C, the crystalline components (e.g., hatrurite, chloromagnesite and quartz) were discarded between its folds. Major components of WTS700 (SiO_2, Al_2O_3 and Fe_2O_3) are 69.71%, while in DaMK are SiO_2 (82.83%) and Al_2O_3 (6%). Accordingly, WTS/DaMK can be used as a pozzolanic material [38].

Table (1): Chemical composition of WTS and DaMK (WT%).

Oxides	WTS 105^{o}C	WTS 700^{o}C	DaMK
SiO_2	40.35	45.72	82.83
Al_2O_3	14.88	19.18	6.00
Fe_2O_3	3.13	4.81	0.50
CaO	5.46	6.8	0.15
Na_2O	0.32	0.2	0.03
K_2O	0.52	0.58	0.05
TiO_2	0.44	0.52	3.4
MgO	0.81	0.86	0.09
MnO	0.28	0.42	0.01
SO_3	1.92	1.16	0.85
P_2O_5	0.16	0.3	0.01
Cl^-	4.67	4.83	0.06
Loss of Ignition (L.O.I)	26.76	14.26	5.84
Total	99.7	99.64	99.82
Molar ratio of SiO_2 /Al_2O_3	4.62	4	23

3.1 Synthesis of WTS/DaMK based geopolymer

In order to study the influence of WTS/DAMK ratio, batches are prepared 75/25, 80/20, 85/15, 90/10 and 95/5 as percentage by mass, respectively. These ratios were chosen to be close to the most suitable ratios of the SiO_2/Al_2O_3 used in forming geopolymer in literature. Many studies deal the ratio between SiO_2/Al_2O_3 which varies from 3 to 5.5, and the ratio between Na_2O/Al_2O_3 varies from 0.8 to 1.2. While the ratio between H_2O/Na_2O ranges from 10 to 14 in some cases according to the potency of each compound at the crystalline state. These ratios were calculated based on the measurements presented in Table 1 and reported in Table 2. The compressive strength of WTS/DAMK geopolymers are illustrated in Fig. 3.

Non-calcined WTS cannot achieve a compressive strength because quartz and crystalline SiO_2 in WTS105 do not develop a geopolymer reaction. Abdelmawla et al. (2019) reported that WTS105 was used as filler only [28]; however, in this study WTS was calcined at 700^{o}C for 1 h with recrystallization and converting the crystalline structure to amorphous oxides that can be calculated in the ratio between SiO_2/Al_2O_3 to distinguish the geopolymerization reaction. WTS700/DaMK85/15 achieves a compressive strength of 8 MPa at 28 days (Fig. 3), although is lower than geopolymers from other aluminosilicate sources [39].

Table (2): Batches of WTS/DaMK based geopolymer with different mixes.

Types of geopolymer	SiO_2/Al_2O_3	Na_2O/Al_2O_3	H_2O/Na_2O	Binder: Sand (by mass)
WTS/DaMK 75/25	5.89	1.24	14.3	1:1
WTS/DaMK 80/20	5.39	1.17		1:1
WTS/DaMK 85/15	5.06	1.15		1:1
WTS/DaMK 90/10	4.70	1.11		1:1
WTS/DaMK 95/5	4.37	1.07		1:1

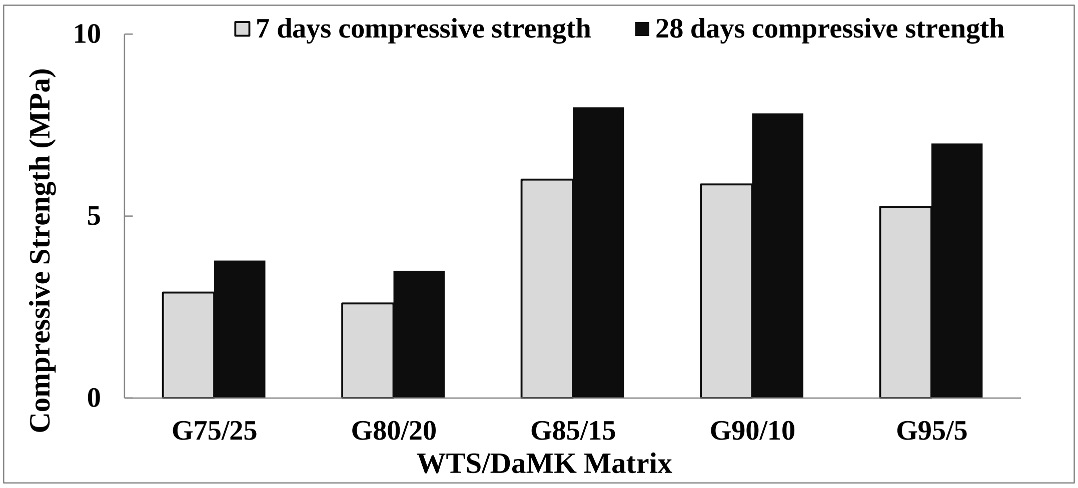


Figure (3): Compressive strengths of WTS/DAMK based geopolymers.

3.2 Efficiency of WTS/DAMK immobilization

The compressive strengths of WTS/DAMK geopolymer (WTS700/DaMK85/15) containing various kinds and concentrations of heavy metals were studied and shown in Table 3. A slight reduction in compressive strength is observed when a heavy metal with a concentration of 300 ppm or less mixed with WTS700/DaMK85/15 geopolymer. Moreover, increased concentration of heavy metals must be calculated to maintain the available strength [23, 24, 40].

Table (3): Batches of WTS/DaMK with different mixes.

Geopolymer matrix	Heavy metal content (ppm)	Compressive strength (MPa)	Immobilization Efficiency (%)
WTS700/DaMK85/15	--	8	--
	Cd 100	7.81	98.83
	Cd 200	7.51	98.56
	Cd 300	7.09	97.82
	Pb 100	7.72	99.57
	Pb 200	7.75	99.48
	Pb 300	7.47	98.93
	Hg 100	7.76	99.43
	Hg 200	7.63	99.38
	Hg 300	7.28	98.92

Batch "WTS700/DAMK85/15-Cd100" to Batch "WTS700/DaMK85/15-Cd300", Batch "WTS700/DaMK85/15-Pb100" to Batch "WTS700/DaMK85/15-Pb300" and Batch "WTS700/DaMK85/15-Hg100" to Batch "WTS700/DaMK85/15-Hg300" incorporating same type and different concentrations of heavy metals. Accordingly, leaching results can be used to distinguish the influence of type of heavy metal ions on the immobilization behaviors on the same basis. The immobilization efficiencies for each batch were measured and reported in Table 3 based on the results in Fig. 4.

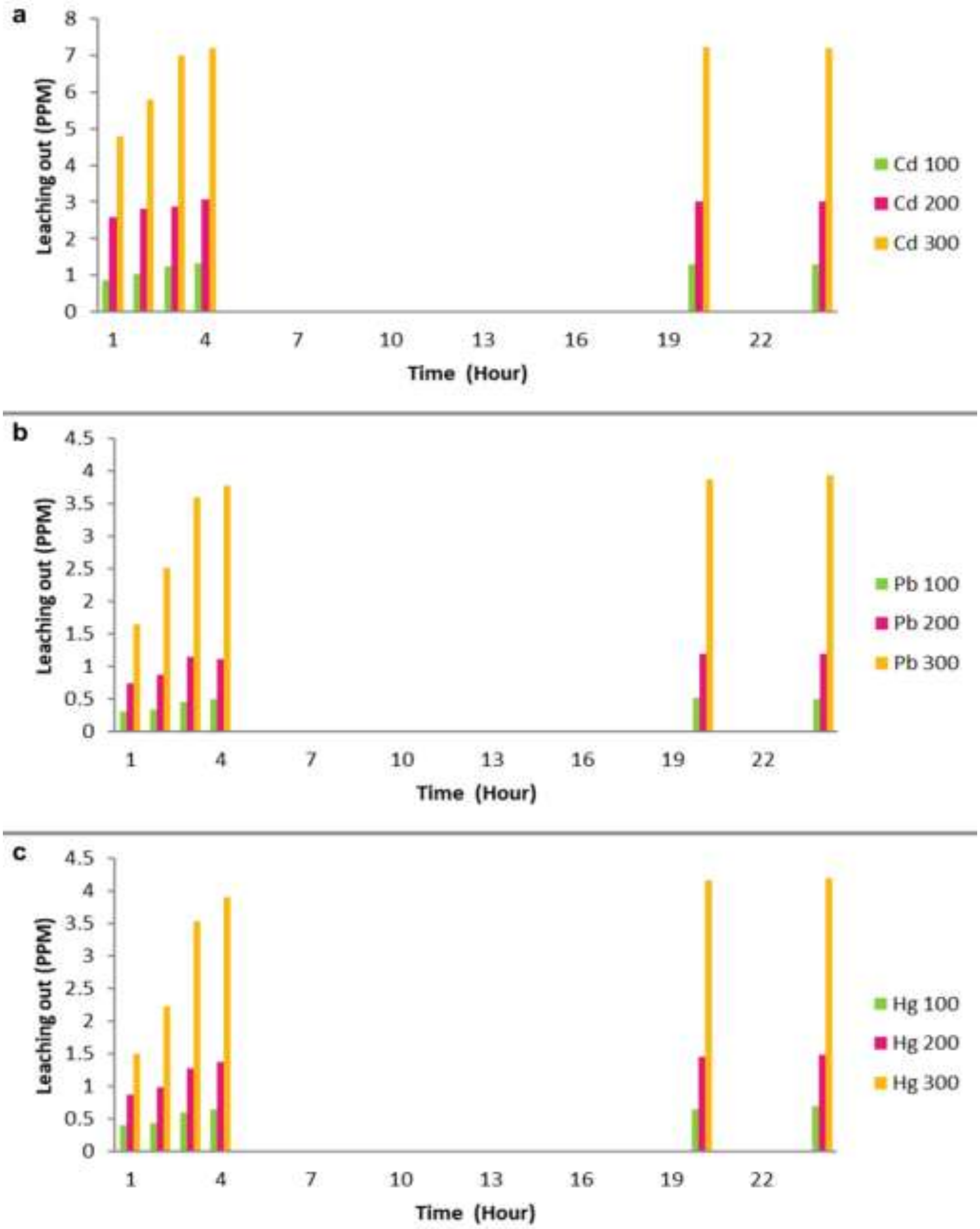


Figure (4): Kinetic leaching curve of WTS/DaMK based geopolymer containing different amounts of Cd^{2+} (a), Pb^{2+} (b) and Hg^{2+} (c).

Leaching of heavy metals started within WTS/DaMK geopolymer with initial concentrations then slightly increased until reaching the equilibrium at four hours (Fig. 4). The WTS/DaMK geopolymer showed high efficiencies with low concentrations e.g., 200 ppm or less, giving negligible concentrations. Therefore, the immobilization efficiency reaches more than 98% (Table 3). The immobilization efficiencies were quickly reduced with adding high heavy metal concentrations (e.g. 300 ppm of Cd^{2+}) which is matching with the compressive strength of same samples in Table 3. We noticed in WTS/DaMK geopolymer matrix that Pb^{2+} and Hg^{2+} ions were leached less than Cd^{2+} in heavy metals with high concentrations regardless the leaching time (Fig. 4).This can be referred to the variability in atomic mass in Pb^{2+} (207.2 u), Hg^{2+} (200.59 u) and Cd^{2+} (112.41 u) [21].

4. CONCLUSION

In this paper, two wastes from water treatment sludge and aluminium sulphate industry were used to produce Geopolymer mortar which consists 85% from calcined WTS (700^{o}C at 1 h) with 15% DaMK achieved a compressive strength of 8 MPa.

Leaching tests showed that the WTS/DaMK geopolymer has a powerful immobilization efficiency of heavy metals Cd^{2+}, Pb^{2+} and Hg^{2+}. The efficiencies reach 98% of heavy metals, incorporated within the WTS/DaMK geopolymer matrix, that have concentrations of 100, 200 and 300 ppm by mass of binders.

We noticed that Pb^{2+} shows a better efficiency of immobilization than Hg^{2+} and Cd^{2+} in case of heavy metals with high concentrations. This can be referred to differences in atomic mass and the chemical interaction with the WTS/DaMK geopolymer matrix.

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