

Removal of Heavy Metals from Wastewater Using Fly Ash Modified Basic Oxygen Furnace Slag Geopolymers

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Abstract—In the current study, fly ash modified basic oxygen furnace slag geopolymers were synthesized and used as geofilters to remove heavy metals from wastewater. Batch experiment based on the removal of heavy metals from synthesized wastewater was conducted so as to investigate the influence of contact time and porosity on the metal removal efficiency and adsorption capacity of the geofilters. Adsorption kinetics was evaluated using Lagergren's pseudo-first-order model, Ho's pseudo-second order and Elovich models. Effect of contact time between geopolymer and wastewater was investigated within the first 15 minutes of adsorption with time interval of 3 minutes. The highest percentages for the removal of Fe^{2+} , Cu^{2+} , Al^{3+} and Ni^{2+} ions with 1% H_2O_2 – FA-BOFS based geopolymer obtained after 15 minutes were 100%, 86.87%, 84.64% and 84.63% respectively. The trend based on metal absorption capacities in an ascending order given as $\text{Fe} > \text{Cu} > \text{Ni} > \text{Al}$. The adsorption capacity of metal within the 15 minutes was relatively constant possibly due to sufficient number of sites upon which metal ion adsorption may have possibly occurred on the outer surface of geopolymers irrespective of H_2O_2 content. Metal ion adsorption rates in the current study strongly adhered to the Ho's pseudo-second-order model based on linear regression fitting method. The correlation coefficients R_2 were 0.999. FA-BOFS based geopolymer can be considered as emergent and versatile material with inherent chemical microstructural property able to remove heavy metals from polluted effluent and metalliferous drainage.

Index Terms—Geopolymer, metal removal, basic oxygen furnace, fly ash.

I. INTRODUCTION

Water pollution due to acidic industrial effluents (wastewater) is now regarded as global crisis [1]. Wastewater is a stream rich of metals formed when water reacts with rocks containing sulfur-bearing minerals [2]. Quality of water has been degraded due to accumulation of metals in the industrial wastewater. Metals are widely used in refineries industries, mining, smelters, foundries, and agricultural activities [2]. Examples of these metals are, to name few nickel, zinc, copper, iron, lead, aluminium, Manganese, Nickel, copper and zinc [1], [2]. Accumulation of metals

from wastewater can be harmful both to the environment and humans through consumption [3], [4]. These metals can be absorbed in human body through consumption of water and result in health effects such as cancer, nervous system damage, and in dangerous cases, death [5]. Environment crisis associated with these metals includes reduction in growth and development of plant like flora and fauna [6]. Hence metals removal from industrial wastewater is of great significant for both sake of human health and environmental conservation.

Several methods for metals removal in the acidic effluents are available in the literature, these include chemical precipitation, coagulation, ion exchange, adsorption, and solvent extraction [6]. Insoluble compounds mainly consisting of metal hydroxi-oxides and/or sulphides are the products of chemical precipitation [7]. This is achieved by reacting soluble/dissolved metals in the solution and precipitant (reagent) to form insoluble solid metal precipitation [8]. The process advantage is that it is simple to carry out and inexpensive equipments are used [8]. The disadvantage of this process lies on that huge amount of chemicals are utilized when reducing the metals into an acceptable discharge range [8]. Large generation of sludge is also major disadvantage in the environment [8]. Ion exchange process is based on the using the ion exchanger resin to attracts the ions [4]. The cations ion exchanger resin will attract the positively charged ions while the anions exchanger resin will attract the negatively charged ions [9]. In many industries, the remaining concentration of ions which are not attracted are carried out in mixed bed vessel where removal of the remaining ions occurs. Ion exchange resin used to attract metal ions are simply an insoluble solid substance capable of attracting these metal ions from any electrolyte solution [4]. The operation of ion exchange is very convenient, and it makes use of cost-effective materials [4]. However, its limitation lies is the inability to handle metals with heavy concentration, this is because suspended solids accumulated in the wastewater may foul matrix [8]. Previous studies shows that only 64% efficiency removal was obtained using nickel and copper aqueous solutions in a batch Ions exchanger [8]. 94% of the removal efficiency was obtained when this method was combined with precipitation [8]-[10]. Adsorption is one of the common methods used in removal of metal ions. An ion should be in aqueous solutions. Adsorption is based on surface phenomenon in which the molecule of adsorbate sticks and thereafter held to the surface of an adsorbent until the state of equilibrium is achieved in the adsorbed molecules [8]-[10]. This process generates a film of the adsorbate on the surface of adsorbent.

Common and widely used adsorbents include activated

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carbon, alumina, and silica.]. More importantly, a wide range of both synthesized and natural zeolites are effectively used for the removal of heavy metals. However, these adsorbents are highly efficient has lately hindered their applicability in the market [10] Hence, the development of adsorbent from upcycled materials such as slags and coal ash has become emergent. Previous studies have shown that iron steel slag and steel making slag as adsorbents resulted in high metal removal efficiency percentages equivalent to 95% of Cu, Fe, Mn and Zn respectively, whereas the use of activated carbons in adsorption has been reported to with the ability to remove 70% Cd, 65% Ni, 60% Mn and Cu 90% [10] Nevertheless, it should be noted that varieties of slags in terms of their chemical compositions confer different physical and chemical properties to the synthesised materials useful for the treatment of polluted wastewater effluent and AMD. Particularly, in the case of BOFS based alkali-system, despite the formation of C-A-S-H and C-S-H, high content of Ca (OH)₂ remains a predominant phase that contributes to the removal of heavy metals by chemical precipitation [11]. In contrast to chemical precipitation, in order to confer zeolite-like adsorption properties to the BOFS based adsorbent, fly ash can be used as an additional source of aluminosilicates in combination with BOFS in order to form a chemically stable, mesoporous and three-dimensional aluminosilicate framework with a predominance of calcium aluminate silicate hydrate endowed with enhanced ion exchange and adsorption property. To the best of the authors' knowledge, the study of metal adsorption on FA-BOFS based geopolymers in a multicomponent solution system has never been investigated. Hence, the aim of the current study is to develop FA modified BOFS geopolymers to use as geofilters for the removal of Fe²⁺, Cu²⁺, Al³⁺ and Ni²⁺ from wastewater. Utilization of BOFS/FA as geopolymer for the removal of metal from wastewater is a key factor toward the valorisation of BOFS and FA raw materials.

II. MATERIALS AND METHODS

A. Materials

TABLE I: FA AND BOFS CHEMICAL COMPOSITIONS

Chemical composition	FA content (% m/m)	BOFS content (% m/m)
Al ₂ O ₃	20.34	4.21
SiO ₂	45.00	10.15
CaO	12.55	47.50
Fe ₂ O ₄	14.60	27.73
CuO	0.01	0.00
MgO	1.60	3.63
Other	5.90	6.78

Fly Ash was obtained from a Camden power station in South Africa. BOFS was collected from ArcelorMittal SA. The major chemical composition of BOFS and FA are shown in Table I respectively. Synthetic wastewater was prepared with a pH around 2.2 to mimic acid metalliferous drainage. H₂SO₄ was supplied by Rochelle chemicals, and it was used to adjust pH of synthetic wastewater. The use of synthetic water is generally accepted as a first step in the determination of the effectiveness of the proposed geopolymer filters [12]. Fe₂SO₄.7H₂O, CuSO₄.5H₂O, Al₂(SO₄)₃.16H₂O and NiSO₄.

6(H₂O) were supplied by Rochelle chemicals and were used to prepare synthetic wastewater. Calibration of AAS instrument was carried out using 1000 ppm standard solutions of Fe, Cu, Al and Ni.

B. Geopolymer Synthesis

The fly ash modified basic oxygen furnace slag geopolymers were synthesized using the author's previous optimization work on the synthesis of FA/BOFS geopolymers [13]. As shown in Fig. 2; FA and BOFS were mixed with a 9 M NaOH solution using a liquid to solid ratio (L/S) of 20%. The H₂O₂ was added into a mixture as a blowing agent to enhance the geopolymer porosity. The content of H₂O₂ was varied from 0.25%, 0.5%, 0.75% to 1% respectively. The mixture was poured into a 50×50×50 mm mould. The mixture was cured at 80 °C for 120 h. Subsequently, synthesised FA-BOFS based geopolymers were washed until neutral pH was obtained before absorption experiment. The washing step ensures that any possible unreacted and free NaOH after geopolymerisation does not lead to chemical precipitation, thus compromises adsorption study by contributing to further metal removal. Physical characteristics profiles of synthesised geopolymers based on the porosity is shown in Fig. 1. It is clearly observed that increase in H₂O₂ content promotes high percentage of porosity within the geopolymer matrix.

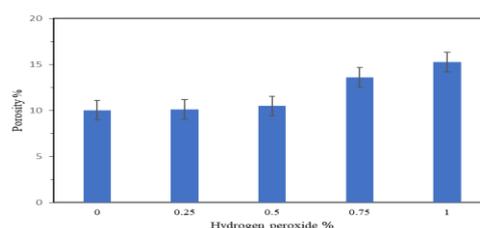


Fig 1. Physical characteristics profiles of FA-BOFS based geopolymer based on porosity.

C. Batch Experiments

The synthesized geopolymers with varied H₂O₂ content were placed in five different beakers respectively to investigate the effect of porosity on the removal of metals. The synthetic wastewater was poured into the beakers using different volumes; 500ml, 1000 ml and 1500 ml respectively. The effect of contact time and between the geopolymer and wastewater on the removal efficiency and adsorption capacity was also investigated. Fig. 2 is a schematic diagram illustrating geopolymers synthesis and batch experiment.

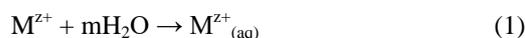


Fig. 2. Schematic diagram of geopolymer synthesis and batch experiment for the removal of metals.

III. RESULTS AND DISCUSSIONS

A. Effect of Contact Time

The effect of contact time on the metal removal efficiency and absorption capacities is shown in Fig. 3 and 4. Results indicate that increase of contact time results to high metal removal efficiency. Time-dependence behavior of most adsorption systems has been reported in various previous studies [12]. The highest percentages for the removal of Fe²⁺, Cu²⁺, Al³⁺ and Ni²⁺ ions with 1% H₂O₂ – FA-BOFS based geopolymer obtained after 15 minutes were 100%, 86.87%, 84.64% and 84.63% respectively. Particularly, complete iron removal was rapidly achieved after 3 minutes. Further increase in time up to 15 minutes had no effect on iron adsorption. The adsorption capacity values were equivalent to 1.08, 0.76, 0.66 and 0.64 mg/g for Fe²⁺, Cu²⁺, Ni²⁺ and Al³⁺ respectively. The trend based on metal absorption capacities ordered from the highest to the lowest value is given as Fe > Cu > Ni > Al. The observed trend can be justified by various factors. According to Fan et al [13], the metal absorption selectivity on the surface of alumino-silicate materials is possibly related to the hydration energy of ions present in the aqueous system [14]. Hydration of ions is referred to as the solvation of cations and anions in water due to H₂O dipole-ionic interactions [15]. General equation for hydration of cations is given in eq. 3.1. Salient consideration is that metal ions with the lowest hydration enthalpy (ΔH_{hyd}) can be easily subjected to dehydration and are therefore readily adsorbed on alumino-silicate materials [14].



Based on the hydration enthalpy present in Table II, it is clearly seen that Fe²⁺ and Cu²⁺ have the lowest enthalpies of hydration equivalent to -1946 and -2100 KJ mol⁻¹ respectively compared to Al and Ni [15]. Hence, it could be observed that Fe²⁺ and Cu²⁺ ions were readily adsorbed in comparison with Al³⁺ and Ni²⁺ ions. Furthermore, previous studies have stipulated that the adsorption selectivity of one metal ions over another mainly depend on the microstructural nature of the absorbent, the ionic radii and electronegativity. Succinctly, the microstructural nature is associated with inherent microporous tetrahedral framework of alumino-silicates which promote sorption of metal ions with smaller ionic radii through the micropores [16], [17]. However, the effect of microstructural nature of FA-BOFS on adsorption capacity is not investigated in the current study.

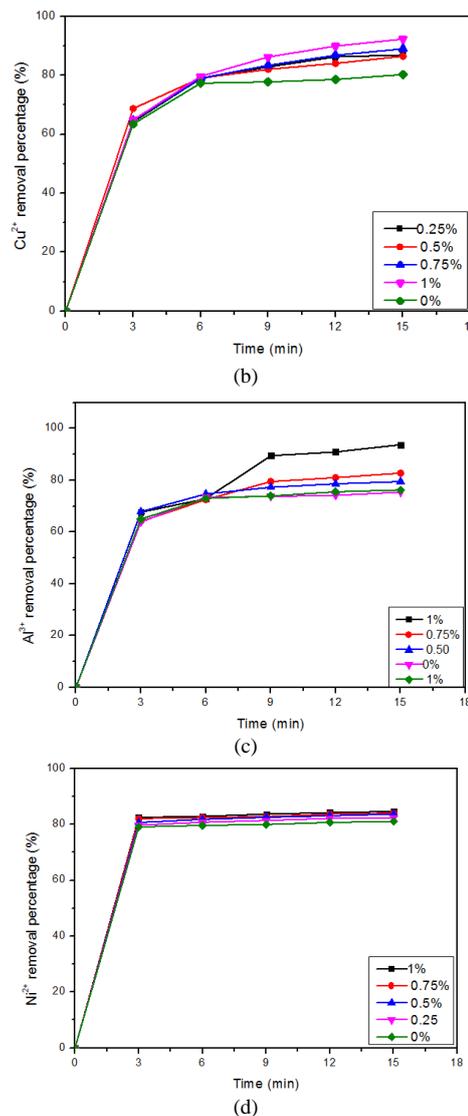
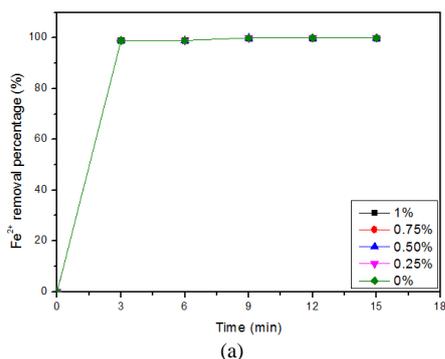
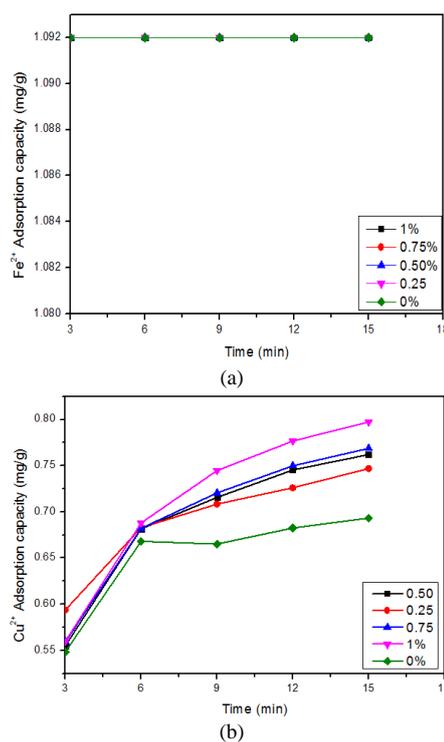


Fig. 3. Effect of contact time on heavy metal ion removal efficiency of FA-BOFS based polymers at different concentrations from 0% up to 1% of H₂O₂ used as a foaming agent at 20°C.



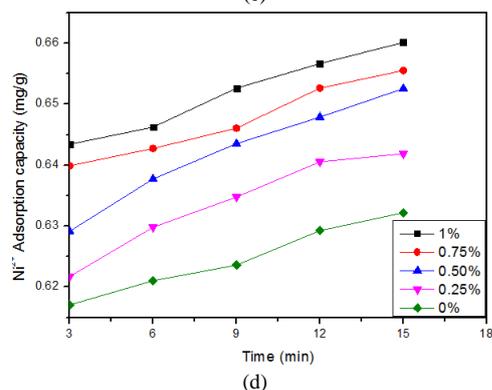
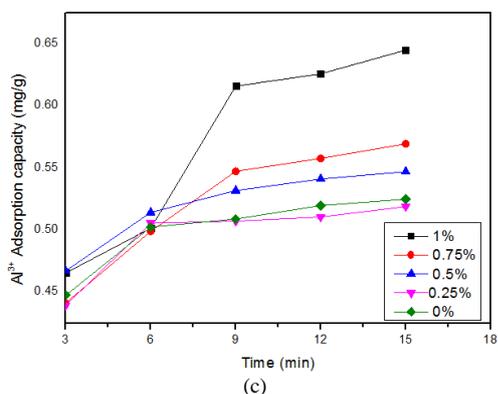


Fig. 4. Effect of contact time on heavy metal ion adsorption capacity of FA-based polymers at different concentrations from 0% up to 1% of H₂O₂ used as a foaming agent at 20°C.

TABLE II: ENTHALPY OF INVESTIGATED METAL IONS

ion	ΔH_{hyd} (KJmol ⁻¹)
Fe ²⁺	-1946
Cu ²⁺	-2100
Ni ²⁺	-2165
Al ³⁺	-4665

B. Effect of Foaming Agent on the Metal Ion Adsorption Capacity

Effect of H₂O₂ content used as foaming agent on metal ion adsorption capacity of synthesized geopolymers is investigated in Fig. 5. Porosity characteristics profile of geopolymers with various concentration of hydrogen peroxide is given in Fig. 1. Experimental observation has been in accordance with the fact that the adsorption capacity of metal within the first 15minutes was relatively constant. Previous studies stipulate that increase in H₂O₂ increase porosity of the geopolymers, therefore the surface area is improved, so is the adsorption rate as the contact time increases. This could only be observed in case of removal of aluminium. In order word, the effect of enhanced porosity is significant as time t increases because porosity improves fluid dynamics within the matrix of geopolymers which facilitates the diffusion of metal and adsorption. However, the general perception is that within the investigated period of 15minutes, it has been suggested that the outer surface of synthesized geopolymers irrespective of H₂O₂ possibly possesses several sites with sufficient energy to remove heavy metal ions by sorption or ion exchange. Hence, metal adsorption capacity is relatively constant within the initial period.

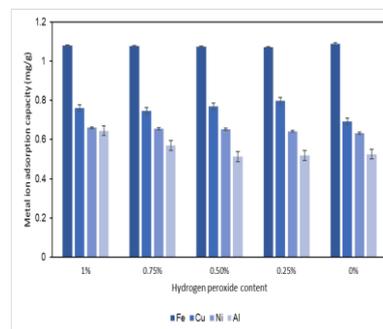


Fig. 5. Effect of foaming agent on the metal ion adsorption capacity after 15minutes at 20°C.

C. Adsorption Kinetics

Insightful information related to the underlying mechanism of sorption systems, rate of reaction, diffusion, mass transfer, surface reaction during adsorption can be obtained through the study of the adsorption kinetics. Adsorption kinetics was studied using Lagergren's pseudo-first order, Ho's pseudo-second order and Elovich models. Table III provides linear forms of first pseudo-first order and second orders as well as the linear form of Elovich model in eq. 2, 3 and 4 respectively. Where q_{eq} and q_t represent the adsorption capacity at equilibrium and time t respectively. Where k_1 and k_2 are the adsorption constants of pseudo-first and second order models respectively. Table IV present all the kinetic parameters and their respective linear regression correlation coefficient R². Results based on R² > 0.99 stipulated that the plot of t/q_t against time t provide the best linear relationship in Fig. 6 (d), (e) and (f), for the adsorption of Cu²⁺, Al³⁺ and Ni²⁺-ions on FA-BOFS based geopolymers with various concentrations of hydrogen peroxide used as foaming agent. This implies that adsorption rates in the current study are governed by the Ho's pseudo-second-order model.

Hence, from the slope of each linear plot, the equilibrium absorption capacity could be obtained in Table IV. The calculated equilibrium absorption capacity for Cu²⁺, and Al³⁺ and Ni²⁺ values were equivalent to 0.837, 0.735 and 0.667mg/g respectively. This implies that the highest metal ion removal efficiency percentages at respective equilibrium time t_e are equivalent to 96.99, 100 and 85.64% for Cu²⁺, and Al³⁺ and Ni²⁺. Particularly in the case of iron ion, adsorption rate was faster, further increase in contact time had no effect. Hence, kinetic modeling of iron adsorption within the investigated time scale is ascertained not to provide information on the iron adsorption rate. Nevertheless, it has been experimentally found that the equilibrium adsorption capacity is 1.08mg/g after 3minutes.

TABLE III: KINETIC MODELS

Linear form equations	
Pseudo-first order	
$\log(q_e - q_t) = -k_1 t / 2.303 + \log q_e$	(2)
Pseudo-second order	
$t/q_t = (1/q_e) t + 1/k_2 q_e^2$	(3)
Elovich model	
$q_t = (1/\beta) \ln(\alpha\beta) + (1/\beta) \ln t$	(4)

TABLE IV: KINETIC MODEL PARAMETERS

Ions	H ₂ O ₂ % in geopolymer	Pseudo-first order			Pseudo-second order			Elovich model	
		k ₁	q _e	R ²	k ₂	q _e	R ²	1/β	R ²
Cu ²⁺	H ₂ O ₂ %								
	1.00	0.048	0.999	0.8639	0.813	0.837	0.9999	0.127	0.952
	0.75	0.104	0.8017	0.9507	1.230	0.798	0.9999	0.092	0.962
	0.50	0.161	0.894	0.9843	0.700	0.854	0.9999	0.129	0.963
	0.25	0.260	0.858	0.9834	0.631	0.892	1	0.148	0.978
0.00	0.264	0.77	0.9887	1.331	0.740	0.9999	0.084	0.786	
Al ³⁺	H ₂ O ₂ %								
	1.00	0.136	0.6951	0.9045	0.652	0.735	0.9959	0.122	0.883
	0.75	0.152	0.5529	0.967	1.277	0.618	0.9997	0.082	0.973
	0.50	0.163	0.55947	0.9767	2.605	0.571	1	0.036	0.536
	0.25	0.031	0.66	0.5714	3.430	0.538	0.9999	0.046	0.766
0.00	0.079	0.5681	0.8269	2.959	0.546	0.9998	0.047	0.892	
Ni ²⁺	H ₂ O ₂ %								
	1	0.118	0.656	0.9620	10.214	0.665	0.9999	0.011	0.909
	0.75	0.187	0.657	0.8706	10.797	0.660	0.9999	0.010	0.852
	0.50	0.081	0.664	0.9944	8.568	0.659	0.9999	0.014	0.989
	0.25	0.046	0.670	0.9635	10.006	0.648	0.9999	0.013	0.987
0.00	0.028	0.671	0.9788	11.830	0.636	0.9999	0.010	0.901	

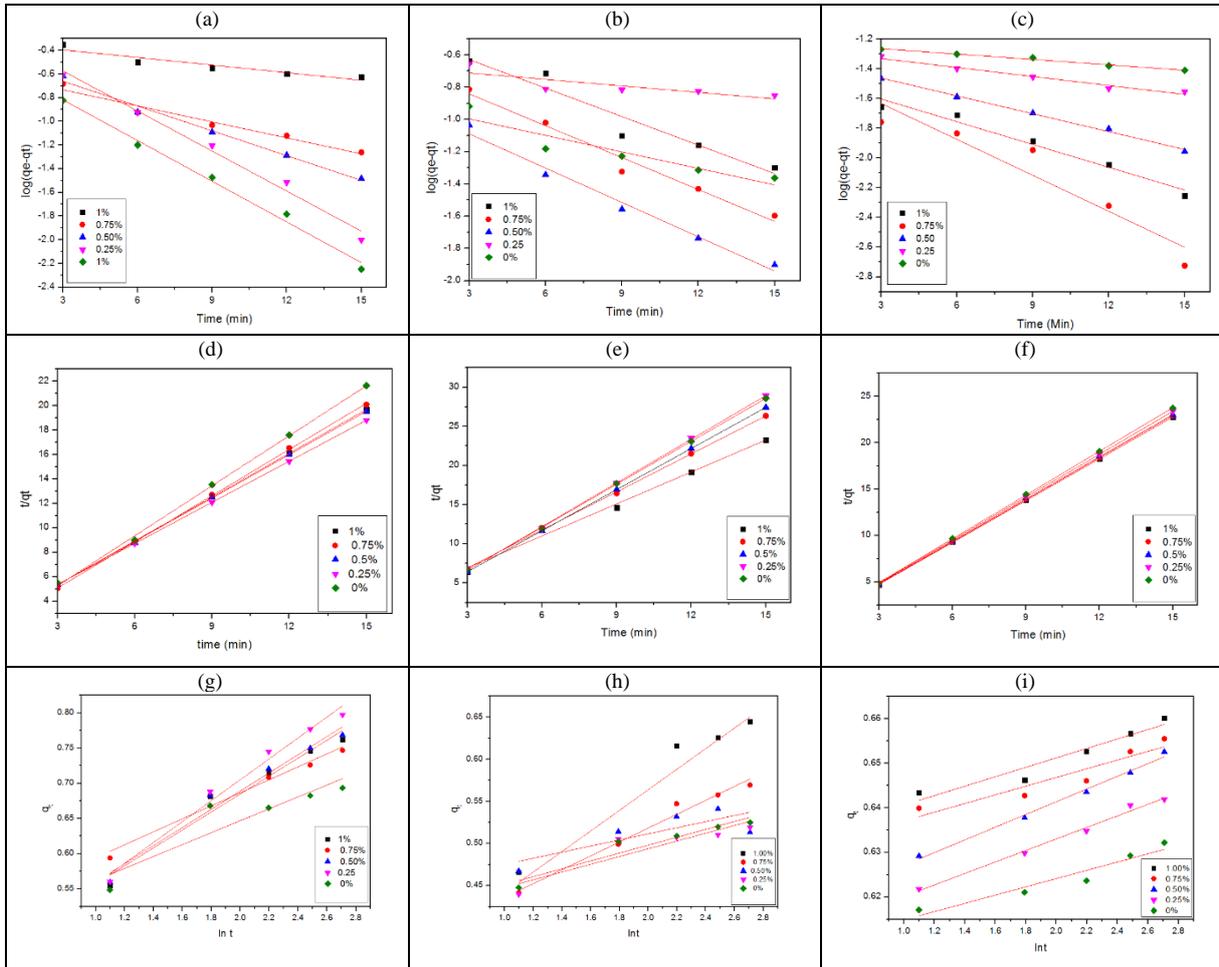


Fig. 6. Absorption kinetic models: (a), (b) and (c) show pseudo-first order plots for Cu²⁺, Al³⁺ and Ni²⁺ adsorption respectively. (d), (e) and (f) show pseudo-second order plot for Cu²⁺, Al³⁺ and Ni²⁺ adsorption. (g), (h) and (i) show the linear plot based on Elovich model for Cu²⁺, Al³⁺ and Ni²⁺ adsorption on FA-BOFS geopolymers respectively.

IV. CONCLUSION

The current study essentially focused on the evaluation of

heavy removals by adsorption using synthesized FA-BOFS based geopolymer. The highest percentages for the removal of Fe²⁺, Cu²⁺, Al³⁺ and Ni²⁺ ions with 1% H₂O₂ – FA-BOFS based geopolymer obtained after 15 minutes were 100%,

86.87%, 84.64% and 84.63% respectively. Adsorption selectivity of one metal ions over another is dependent on the enthalpy of hydration of metals, the microstructural nature of the geopolymers, ionic radii and electronegativity. Ascending order of adsorption capacity of synthesized geopolymers is given as: Fe<Cu<Ni<Al. Within the 15 minutes, adsorption capacity of geopolymers with various concentration of H₂O₂ used as a foaming agent was relatively constant possibly due to existence of sites on the outermost exposed layer of geopolymers inholding sufficient energy to impart metal adsorption irrespective of enhanced porosity. Investigated Adsorption rates of the heavy metal ions strongly adhere to pseudo-second order model developed by Ho.

Based on these appreciable results, it is ascertained that FA-BOFS based geopolymer can be considered as a promising and versatile material useful for the treatment of polluted effluent and acid metalliferous drainage.

CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHOR CONTRIBUTIONS

N.T. Sithole conducted the research; J. M. Nseke analyzed the data; All authors wrote and reviewed the paper; all authors had approved the final version.

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Thandiwe Sithole holds a PhD in chemical engineering obtained at the age of 28, post graduate diploma in higher education and a certificate of a emerging leader. She is currently registered for a master's in business administration (MBA) at University of Suffolk (UK). She is a senior lecturer at the Department of Chemical Engineering, Faculty of Engineering and the Built Environment, University of Johannesburg. She is registered with

the Engineering Council of South Africa. She has filed one patent and authored/co-authored over 25 peer reviewed publications. She has supervised over 30 undergraduates and postgraduate students. Her mentorship and research experience is enviable as she guides her both undergraduate and postgraduate level students through the research journey.

Her research interest is in the field of investigating possible solutions that will eradicate problems associated with Environmental pollution such as industrial solid waste and wastewater. Her research in the field of industrial solid waste involves beneficiation of such solid waste materials by developing valued added products that can be used in building and construction with the purpose of commercialisation. She also conducts research in the field of Wastewater treatment, 3D printing in building and construction, geopolymerisation from waste.

Dr. Sithole is a non-Executive director and a technical advisory board chair at The Platinum Incubator (TPI). She was one of the nominees for the NRF award (research excellence award for next generation researchers 2019). She was one of the two South African young scientists selected by DST to represent South Africa in the Africa-China young scientist Inn tour. She served in the Women in Engineering and Built Environment (UJ) and the Transformation committees. She is also a recipient of the National Research Foundation (ZA) grants.



Joseph Nseke got a bachelor of technology degree holder from Vaal University of Technology (VUT) and is currently pursuing his master's degree in metallurgical engineering at the University of Johannesburg (UJ). He is a registered engineering technologist at the Engineering Council of South Africa (ECSA) and work as a technician in the Extraction Metallurgy laboratory at the University of Johannesburg. He is proficient in the use of statistical

analysis and Design of Experiment (DoE) based tools and knowledgeable in advanced analytical techniques such as Fourier transform spectroscopy, nuclear magnetic resonance, scanning electron microscopy, inductively coupled plasma, X-ray diffraction and X-ray fluorescence spectroscopic techniques. He has brought a resourceful input to various undergraduate and postgraduate research projects in the department of metallurgy and chemical Engineering, faculty of engineering and built environment at the University of Johannesburg and co-supervised different undergraduate research projects.

Nseke's research interest is merely but not exclusively focused on hydrometallurgy and mineral processing. Several investigations conducted by him intend to essentially contribute to the improvement of metal recovery through hydrometallurgical leaching processes including chemical separation methods such as solvent extraction, ion -exchange, cementation, chemical and physical precipitation methods.