

Geopolymer Composites Designed for Water and Wastewater Treatment

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Definition

Since the discovery of geopolymers four decades ago, they have come quite a long way successfully and gained attention, mostly due to the effortlessness for geo-synthesis with lesser emission of greenhouse gases (GHG). Their admirable properties, such as toughness, heat and fire resistance, refractory nature and radiation hardness, altogether make them promising for applications for radioactive waste containment, as well as for pozzolanic action, making them multi-functional. In realism, lots of industrial materials are derived using geopolymers. For example, fiber-based geopolymer composites are regarded as fire-resistant. Quite a lot of geopolymeric composites are positioned in metal tool coatings and the construction of cabinets of airplanes and buildings, with a view to trim down the intensity of inferno incidents. The polymeric chain-like structure of geopolymers contributes higher chemical resistance, lower shrinkage and enhanced resistance to abrasion with high early mechanical strength. This entry describes that applications and interaction of geopolymer.

1. Applications of Geopolymers for Water and Wastewater Treatment

Nowadays, geopolymers are regarded as versatile materials, which have drawn attention in scores of environmental applications, and hence, are being employed nearly in all fields of technology, for instance, in water and wastewater treatment as adsorbents or ion-exchangers, high-pressure membranes and filtration media, photo-catalysts, anti-microbial materials, pH buffers, carrier media in bioreactors and for the solidification or stabilization of water and wastewater treatment residues. The applications are not merely those; they can be employed as anti-bacterial materials following their incorporation with copper (Cu) or silver (Ag) in the course of ion exchange or supplementation of nanoparticles, namely, silver-silica nano-composites into the geopolymeric alumina-silicate matrix ^{[1][2][3]}. Thus, they are useful as anti-microbial binders with analogously modified plentiful zeolites to designate their potential usage for the disinfection of wastewaters or sub-surface water ^{[4][5][6]}.

Since the discovery of geopolymers four decades ago, they have come quite a long way successfully ^[7] and gained attention, mostly due to the effortlessness for geo-synthesis with lesser emission of greenhouse gases (GHG) ^[8]. Their admirable properties, such as toughness, heat and fire resistance, refractory nature and radiation hardness, altogether make them promising for applications for radioactive waste containment, as well as for pozzolanic action, making them multi-functional. In realism, lots of industrial materials are derived using geopolymers. For example, fiber-based geopolymer composites are regarded as fire-resistant. Quite a lot of geopolymeric composites are positioned in metal tool coatings and the construction of cabinets of airplanes and buildings, with a view to trim down the intensity of inferno incidents ^[9]. The polymeric chain-like structure of geopolymers contributes higher chemical resistance, lower shrinkage and enhanced resistance to abrasion with high early mechanical strength ^[10].

At present, geopolymers are an emerging class of materials for the eco-benevolent refurbishment of decrepit infrastructures, recovery of marsh environments and sustainable reinforcement of structural amenities. The highly workable properties of geopolymers, such as water retention aptitude, when owning shear stress of ~80 Pa at a shear rate of 110 s⁻¹ and compressive strengths of ~40 MPa at 7 days curing, facilitate the tailoring of them. Furthermore, they possess attributes that make geopolymers a significant class of promising materials for the containment and capping of nuclear wastes perils ^[11]. Moreover, the development of geopolymer sorbents necessitates considerations for how to employ them in general

practice. It is feasible to dose powdered sorbent constantly; however, then an added process phase is needed to separate the utilized sorbent, and operational costs may go higher. As a result, a choice of alternative methods is accessible to manufacture highly porous and permeable geopolymers, e.g., direct foaming, sacrificial template method, freeze casting, granulation or additive manufacturing (AM), i.e., 3D printing.

1.1. Geopolymers as Adsorbents or Ion-Exchangers

The key drivers for this emerging approach for the application of geopolymers as adsorbents or ion exchangers are their outstanding mechanical and chemical stability coupled with a fairly uncomplicated, lower carbon, low-energy, user-friendly and cost-effective manufacturing process of geopolymerization [12]. There exist numerous efficient and familiar wastewater treatments to get rid of heavy metal cations present in it, including adsorption [13][14][15], ion-exchange, photo-catalytic degradation, i.e., as photo-catalysts, membrane filtration (or separation) materials, chemical precipitation, bio-remediation sedimentation, pH adjustment agents, the solidification or stabilization of water treatment residues reverse and forward osmosis [16][17][18][19][20][21][22][23][24][25][26][27][28][29][30]. Consequently, geopolymers offer viable alternatives to competing materials for water and wastewater treatment, such as conventional ceramics or synthetic zeolites, polymeric components in terms of cost, an assortment of choice, eco-impacts, and of course, performance.

In recent times, on account of the significant attributes, such as a simple method, low-cost, environment-friendly nature, higher competence and mild micro-spheres synthesis method, geopolymers are successfully developed and commonly used as adsorbents, inorganic membranes, catalysts and for the immobilization of dangerous metal ions. Adsorption is regarded as the most efficient, uncomplicated and widespread technique for water decontamination [31][32][33][34][35]. Undeniably, activated carbon is the standard adsorbent material and is well-known as the most utilized adsorbent for water and wastewater treatment with an excellent demonstration of higher adsorption competencies for the removal of heavy metal cations, but its use gets hindered due to its comparatively high cost [36]. Hence, in spite of the exceptional adsorption ability of this material, its higher production costing is the root cause of wide-ranging research for low-cost yet effective options in recent times. Significantly, the unique porous structure of geopolymers and the existence of negative charges on the aluminum-tetrahedra of the resulting gels, they can efficiently and successfully absorb metal ions, namely, Pb^{+2} , Cu^{+2} , Cd^{+2} , Mg^{+2} , Hg^{+2} , Cr^{+3} , etc. [37][38][39][40][41][42].

The performance of heavy metal adsorption by geopolymers relies mostly on the structures formulated by the geopolymer gels; nevertheless, owing to the presence of numerous inert crystalline phases in them, the clear-cut correlations among the geopolymeric gels and performance of adsorption by them cannot be verified all the time. The earlier research works deal with the hydrochloric acid (HCl) dissolution to verify the degree of reaction of geopolymerization, and as inert crystalline phases are insoluble in acid, the gels and other dissolved matters can be measured based on the mass alterations. The adsorbents in powder form may necessitate the support materials use, such as porous ceramics and polymer foams, [42] to permit their industrial utilization or a separation step, such as pressure filtration after wastewater treatment course of action, both being harmful to the wastewater treatment costing besides the rising complexity of the procedures. The geopolymers can fruitfully be used as green adsorbents or ion exchangers by manufacturing them with wastes as precursors extending a systematic solution for their disposal too. Thus, the adsorption using these environ-benign adsorbents is a superb and very much progressive technique for the elimination of not merely inorganic but also organic contaminants from aqueous mediums, particularly through geopolymers, which can be produced employing solid wastes as precursors [43].

The ever-increasing diverse wastes from various fields, such as fly ash, metakaolin or low-calcium alumina-silicate solid wastes, accumulating as landfills are not only hazardous to the environmental sustainability but are also resource-wasting and polluting water, soil, etc., with lots of negative impacts

on the health of living beings, and can advantageously be used as precursor resources. For instance, fly ash (FA) is being used as higher value-added applications, viz., the synthesis of FA-ZnO enclosing nanofibers for adsorption and photo-catalytic degradation of organic dyes, etc. [44]. Furthermore, the production of geopolymers enables benefiting industrial, agricultural, etc., wastes containing alumina and silica to be utilized as a resource for precursors, such as fly ash, clay, slag, raw kaolin, metakaolin and rice husks. Lots of research works used rice husk as a precursor, considering it as a silicon source useful for preparing a range of geopolymers making the costing comparatively low. Moreover, their uses for disinfection made ground, gaining the geopolymer technology substantial attention from not only researchers but also industries for concentrating on cleaner manufacturing. This is an additional advantage to offer systematic disposal of all such kinds of wastes that can be used to design a geopolymer useful for water and wastewater treatment and reclamation as well [45]. These compounds raise quite remarkable questions about their surface and structure, e.g., the almost total exclusion of diffusion of sequestered metal ions and avoidance of issues linked to leaching of geopolymer adsorbents [46].

As referred, geopolymers are famous for occupying inter-crossed linked bonds with cationic ends on the surface, structurally permitting the entrapment of toxic and radioactive metals through charge balancing. On the other hand, the micro-structure of geopolymer in the nano-metric scale of 5 to 10 nm frequently consists of numerous pores inside a highly porous -Al-O-Si- repetitive unit, and these voids are probable to provide room for ionic integration, replacement and balances too. Extraordinarily, geopolymers own a key structural unit analogous to zeolites; Provis et al. [47] unveiled that they possess an amorphous gel phase and nano-crystalline zeolites agglomerates. According to them, a proposal has been put forward that the geopolymers and zeolites could be inter-converted under suitable conditions. Thus, the conversion of geopolymers into zeolites is regarded as an exceptional strategy since the well-organized recycling of solid wastes from industries and the economical production of costly zeolites can be recognized concurrently. Beforehand, Cui et al. [48] authenticated using a high-resolution transmission electron microscope and particular area electron diffraction that numerous nanometer-ordered structures were detected in the amorphous geopolymer ($\text{Al}_2\text{O}_3\text{2SiO}_2\text{Na}_2\text{O7H}_2\text{O}$), successfully acquiring the Na-A type molecular sieve from this geopolymer through a hydrothermal reaction. Afterward, a series of sodium (Na)-zeolites, including sodium (Na)-A, faujasite, sodium (Na)-P and sodium (Na)-X were attained via the hydrothermal alteration of geopolymers when sodium cations (Na^+) were present, which displayed huge potential for membrane separation and bulky adsorbent utilizations. It is eminent that the development and nucleation of zeolites rely upon the concentration of alkali metal cations and their kind [49][50][51][52][53].

The zeolites, which are derived from geopolymers, are acquired from a solution of Na^+ cations resulting in restraining their diversity. Hence, it is imperative to develop an extensive variety of zeolites from geopolymers for utilizations linking bulky adsorption and membrane separation. On the other hand, in a research study, Rossi blended a biomass fly ash as 75% with 25% geological natural metakaolin and used the mixture as a precursor for geo-synthesis through geopolymerization and achieved an end-product with a huge specific surface area of $56.35 \text{ m}^2/\text{g}$ together with a brilliant pressure resistance of up to $\sim 10 \text{ MPa}$. Furthermore, a noteworthy improvement in adsorption competence and compressive strength was monitored in a study whereby the supplement of cork waste residue was made into metakaolin-based geopolymers [54]. Moreover, the existence of dyes in wastewaters is considered a somber crisis for ecology [55]. Cation-enclosing dyes, such as methylene blue or methylthionium chloride with a formula $\text{C}_{16}\text{H}_{18}\text{ClN}_3\text{S}$, is a salt that is cationic and used as a dye, mostly in the textile industry for cotton, wool and leather dyeing, paper and plastics, as well as furniture coloring. Methylene blue is very injurious to the health of living beings since it can cause blindness, allergy, abdominal disorders, asthma, etc. [25][55]. As a result, such dyes must be removed from aqueous solutions by means of ultra-filtration, photo-degradation or ion-exchange methods. Still, adsorption is one of the most efficient and affordable routes for water and wastewater treatments [56][57]. For the reason that it is a cationic dye, hence, positively charged; the application of geopolymers with negatively charged networks as adsorbents is the most

promising solution in this context. Furthermore, the likelihood of the development of zeolites in the course of the geopolymerization of metakaolin can optimistically influence the procedures of adsorption.

The zeolite is portrayed by a higher selectivity headed for methylene blue [58]. The few modern research works are throwing light on the application of zeolite-type geopolymeric green nano-adsorbents considering their higher effectiveness, steadiness, trouble-free production system, exceptional binding characteristic and attractive lower costing [14][58][59][60][61]. They are found highly porous and can be manufactured at as low as room temperature through a simple process of geopolymerization among aluminum and silicon sources in an alkali medium [14][60]. Zeolites resembling geopolymers are appreciably useful for a variety of eco-friendly applications, viz., the taking away of heavy metals and photo-catalytic degradation of a few organic compounds, such as benzophenone, metronidazole, p-nitrophenol and eriochrome black T [62][63][64][65][66][67], along with some significant industrial uses, namely, diversion of methanol (CH₃OH) to olefins and phenol hydroxylation for water and wastewater treatment.

1.2. Thermo-Dynamics of Adsorption

The necessary data with regard to the nature and thermo-dynamic viability of the adsorption progression can be obtained with the help of the thermo-dynamic parameters, standard change of entropy (DS₀), change of standard free energy (DG₀) and change of standard enthalpy (DH₀). In the context of geopolymer adsorbents, the accounted DHO values of adsorption are commonly found as positive [68][17][28], designating with respect to the adsorption competence that it escalates at elevated temperatures. Nevertheless, one differing upshot as DH₀ 10 kJ/mol was found reported by Li et al. [69] in the case of elimination of methylene by fly ash-based geopolymer. This is additionally supported by the fact that the values of DS₀ are constantly positive, signifying that the system entropy enhances are subsequent to the adsorption course of action [28][69][70]. This can be comprehended with the separation of hydrated water molecules from the metal ion, i.e., inner-sphere complexes are formulated, and specific adsorption takes place previous to the attachment to the surface sites of geopolymers, which is regarded as an energy-requiring procedure [68][28][70].

1.3. Adsorption of Radioisotopes

Fantastically, geopolymer composites are employed to adsorb a number of elements that have radioisotopes, viz., caesium (Cs), strontium (Sr), radium (Ra) and cobalt (Co). The elimination of radioisotopes from water has turned out to be progressively more significant in the aftermath of the incident of the accident at Fukushima nuclear site, Japan, in 2011 [71]. In the aqueous environment, caesium, which is highly soluble and stable, occurs as Cs. The radioactive isotope ¹³⁷Cs is of great concern because it has a long half-life of 30.2 years. On the other hand, strontium is found present chiefly as Sr²⁺ in the aqueous environment and possesses quite a lot of radioactive isotopes. Of which, ⁹⁰Sr with a half-life of 28.9 years is the most noteworthy [72]. Usually, all radium (Ra) isotopes are found present as Ra²⁺ in low salinity conditions with half-lives ranging between a few days and 1600 years. The metakaolin-based geopolymer is potentially effective for the adsorption of the caesium (Cs), and therefore, preferable to lead (Pb²⁺), copper (Cu²⁺), cadmium (Cd²⁺), nickel (Ni²⁺) and zinc (Zn²⁺). The adsorption competence is not influenced by the presence of sodium chloride (NaCl) up to 10 weight-% concentration. Chuang and Liao [73] adsorbed caesium (Cs⁺) ions by phosphate-based geopolymer enclosing potassium (K⁺), zinc (Zn²⁺) cations and Ferrocyanide anion [(Fe(CN)₆)⁻⁴]. Lee et al. [74] utilized porous geopolymeric blocks manufactured with fly ash and blast furnace slag with a view for eliminating Cs⁺. Geopolymer composites are compared satisfactorily with so many other types of adsorbents for the removal of Cs⁺, which show the highest adsorption competence [71]. Chen et al. [75] investigated the elimination of Cs⁺, Sr²⁺ and Co²⁺ ions by metakaolin and fly ash-based geopolymer composites and achieved very high adsorption competencies. Moreover, it is possible to adsorb these ions with 0.1 M HCl, which suggests that the adsorbent could be regenerated [75]. Geopolymer composites using fly ash are reported to be potentially efficient for taking away cobalt (Co²⁺). At this juncture, it is interesting to note that both the radioisotopes and non-radioactive isotopes exhibit analogous aqueous chemistry. In the end,

radium isotopes can be significantly removed by foamed geopolymers where there is an incorporation of barium sulfate (BaSO₄), and the material is suggested to be employed as a passive filtration material. Thus, geopolymer composites can brilliantly remove different polluting ions efficiently.

2. Geopolymer Interaction Mechanism with Heavy Metals

Geopolymer removes heavy metals via an adsorption process in which heavy metal ions, or adsorbates, cling to the accessible binding surfaces of geopolymer. The adsorption isotherm is required to explore the nature of the interaction between geopolymer and heavy metal ions and to improve the application. In this scenario, the Langmuir and Freundlich models are typically used. **Table 1** shows the adsorption of metals through a geopolymer and **Table 2** represents adsorption of dyes in different types of geopolymers. Ge et al. [76] also showed that when MK-based porous geopolymer spheres were employed to remove Cu²⁺ the Langmuir model fit better than the Freundlich model. These data imply that Cu adsorption by FA-based standard geopolymers is monolayer. In the instance of Pb²⁺ removal, FA-based traditional geopolymers and found that the Langmuir model is more suited to represent the adsorption process than the Freundlich model [77]. As a result of the superior match of the Langmuir model seen in most studies, it is possible to conclude that heavy metal ion adsorption onto the geopolymer adsorbent is classified as monolayer. The interaction between Cu²⁺ and FA-based conventional geopolymer within a temperature range of 25–45°C using both the Langmuir and Freundlich models and found that both models had a good correlation coefficient, however the Langmuir model fit better [76]. The removal effectiveness of Ni²⁺ using LD-slag based traditional geopolymer and discovered that the Langmuir model suited better than the Freundlich model, implying that the adsorption is monolayer adsorption [70]. On the other hand, classified the adsorption process based on the kind of bond produced, which are physisorption and chemisorption [78]. The former is preferable because the weak Van der Waal force allows for easy renewal of geopolymer as an adsorbent by simple or steam washing, chemical or thermal treatment. It has been observed that after 6–10 consecutive times of use, the adsorption capacity of geopolymer was lowered by roughly 1–10%. The latter, on the other hand, was observed to occur in the majority of the investigations, which is related to the fitting of the Langmuir isotherm. Fourier-transform infrared spectroscopy (FTIR) was used to understand the adsorption mechanism at the microscopic level, despite the fact that very little literature was available. Typically, wavenumbers ranging from 400 or 450 cm⁻¹ to 4000 cm⁻¹ were used. Furthermore, no significant change in the absorption band of Al–O–Si bending vibrations and Si–O–Si bending vibrations was seen following heavy metal adsorption [79]. However, following Mn²⁺ and Cu²⁺ adsorption, the adsorption band of H–O–H bending vibration at 1647 cm⁻¹ was relocated to 1637 cm⁻¹, and the peak at 1450 cm⁻¹ was definitely shifted to 1431 cm⁻¹ after Cu²⁺ adsorption. These spectral changes suggested that when Mn²⁺ and Cu²⁺ were attached to MK-based conventional geopolymer, a complexion with –OH groups occurred. When Ni²⁺ was adherent to LD-slag based conventional geopolymer, the peak of the spectra at 963 cm⁻¹ got broader, and postulated that this was due to the formation of a metal ion layer surrounding the LD-slag based conventional geopolymer matrix. Another tiny signal was found at 1447 cm⁻¹, showing that heavy metal ions may be chemically bound to the LD-slag-based conventional geopolymer matrix [70].

Table 1. Adsorption of metals through a geopolymer.

Source Material for Geopolymer	Adsorbate	Alkaline Activator	Adsorption Capacity (mg/g)	References
Metakaolin, Rice Husk Ash	Crystal Violet	KOH	276.9	[80]
Fly Ash	Cd ²⁺	NaOH, Na ₂ SiO ₃	9.02	[81]
Pyrophyllite	Cd ²⁺	NaOH	7.82	[82]
Pyrophyllite	Co ²⁺	NaOH	7.1	[82]
Metakaolin	Co ²⁺	NaOH, Na ₂ SiO ₃	69.23	[79]
Metakaolin	Mn ²⁺	NaOH, Na ₂ SiO ₃	72.34	[79]

Source Material for Geopolymer	Adsorbate	Alkaline Activator	Adsorption Capacity (mg/g)	References
Pyrophyllite	Ni ²⁺	NaOH	7.28	[82]
Pyrophyllite	Pb ²⁺	NaOH	7.54	[82]
Ld Slag	Zn ²⁺	NaOH, Na ₂ SiO ₃	86	[83]
Fly Ash, Blast Furnace Slag	Cs ⁺	NaOH,	15.24	[74]
Metakaolin	Cu ²⁺	KOH, silica fume	40	[84]
Metakaolin	Cu ²⁺	NaOH, Na ₂ SiO ₃	62.5	[85]
Metakaolin	Ni ²⁺	NaOH, Na ₂ SiO ₃	42.61	[79]
Blast Furnace Slag	SO ₄ ²⁻	NaOH, Na ₂ SiO ₃	119	[28]
Metakaolin	Cd ²⁺	NaOH, Na ₂ SiO ₃	98.10	[86]
Metakaolin, Clinoptilolite	Cr ³⁺	NaOH, Na ₂ SiO ₃	21.84	[86]
Fly Ash, Iron Ore Tailings	Cu ²⁺	NaOH, Na ₂ SiO ₃	113.41	[18]
Metakaolin	Cu ²⁺	NaOH, Na ₂ SiO ₃	44.73	[86]
Metakaolin, Clinoptilolite	Pb ²⁺	NaOH, Na ₂ SiO ₃	261.22	[86]
Volcanic Tuff	Zn ²⁺	NaOH	14.83	[87]
Metakaolin, Clinoptilolite	Zn ²⁺	NaOH, Na ₂ SiO ₃	35.88	[86]
Metakaolin, Al ₂ O ₃	Methylene Blue	H ₃ PO ₄	4.26	[25]
Metakaolin	Ca ²⁺	NaOH	24	[88]
Fly Ash	Cu ²⁺	NaOH	152.3	[68]
Metakaolin	Cu ²⁺	NaOH	34.5	[88]
Metakaolin	Pb ²⁺	NaOH	45.1	[88]
Metakaolin	Cu ²⁺	NaOH, Na ₂ SiO ₃	52.63	[76]
Fly Ash	Methylene Blue	NaOH, Na ₂ SiO ₃	50.7	[89]
Metakaolin	NH ₄ ⁺	NaOH, Na ₂ SiO ₃	21.07	[90]
Blast Furnace Slag	As(III)	NaOH, Na ₂ SiO ₃	0.52	[22]
Fly Ash	Co ²⁺	NaOH, Na ₂ SiO ₃	66	[20]
Fly Ash	Co ²⁺	NaOH, Na ₂ SiO ₃	59	[91]
Fly Ash	Co ²⁺	NaOH, Na ₂ SiO ₃	52	[91]
Fly Ash	Cu ²⁺	NaOH, Na ₂ SiO ₃	77	[20]
Blast Furnace Slag	Ni ²⁺	NaOH, Na ₂ SiO ₃	4.42	[22]
Fly Ash	Pb ²⁺	NaOH, Na ₂ SiO ₃	118.6	[17]
Fly Ash	Pb ²⁺	NaOH, Na ₂ SiO ₃	6.34	[92]
Blast Furnace Slag	Sb(III)	NaOH, Na ₂ SiO ₃	0.34	[90]

Source Material for Geopolymer	Adsorbate	Alkaline Activator	Adsorption Capacity (mg/g)	References
Metakaolin	NH ₄ ⁺	NaOH, Na ₂ SiO ₃	32	[93]

Table 2. Adsorption of dyes in different types of geopolymers.

Geopolymer as a Adsorbent	Dye	Adsorption Capacity (mg/g)	Efficiency Degradation (%)	References
TiO ₂ geopolymer composite	MB	20.11	97	[94]
Phosphoric acid-based geopolymer	MB	3.01		[25]
Fly ash geopolymer	MB	37.04	-	[95]
Metakaolin-based geopolymer	MB	43.48	-	[96]
Magnetic geopolymer	AR97	1814.27		[97]
Geopolymer	CR	-	100	[98]
Fly ash-based geopolymer	BY	36.364		[99]
Magnetic geopolymer	AG	183.17		[100]
Metakaolin-based geopolymer	MV10B	276.9		[80]
Metakaolin geopolymer	MO	0.333	-	[101]
Magnetic geopolymer	PR	39.21		[100]
Alkali-activated phosphorous slag	BV	46.58		[102]
Fly ash geopolymer	MB	-	92.79	[103]
Alkali-activated phosphorous slag	MGO	46.36		[102]
Magnetic geopolymer	AG16	400		[104]
Geopolymer	MV	-	91.16	[105]

Methylene blue—MB, Basic yellow 2—BY, Acid green—AG, Procion red—PR, Basic violet—BV, Malachite green oxalate—MGO, Acid green 16—AG16, Methyl violet 10B—MV10B, Acid red 97—AR97, Methyl orange—MO, Crystal violet—CV, Congo red—CR, Methyl violet—MV.

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