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Synthesis of zeolite from marble powder waste: a greener approach and its application for the removal of inorganic metals from wastewater

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ABSTRACT

The wastewater containing heavy metals, produced at various stages of operation in textile, printing, plastic, and paper-making industries, pose major hazards to the environment and the public health. Therefore, it is necessary that the pollutants should be treated before discharge by using highly efficient adsorbents. Zeolite is a potential material and can conveniently be processed as adsorbents for the removal of environmental pollutants. A wide range of commercial zeolite has been marketed but due to high cost it is of limited use. The present research offers a green approach for the application of zeolite synthesized by using marble powder waste as a precursor. The significance rests on the conversion of waste into a useful adsorbent marble zeolite (MZ). Zeolite is an environmental friendly, novel, and cost-effective adsorbent. In the current study, remediation of heavy metals in industrial wastewater is done using zeolite. The marble waste contains metals like Zn, Ni, Pb, Cr, Cd, and Cu in it and this waste itself also used as an adsorbent for removal of metals and its efficiency is ≥85%. The batch method has been employed for metal removal using different standard solutions at concentrations of 50–200 mg/L of individual metals (Zn, Ni, Pb, Cr, Cd, and Cu). The optimum removal efficiency is determined at 50 mg/L and the results depicted the performance of MZ (1 mg/kg) in removing inorganic pollutants upon adsorbate–adsorbent contact for 30 min, at lower dose of MZ. It was also found that the higher the induced concentration, the lesser the removal efficiency. It may be due to the limiting factor of adsorbent dose. The adsorbent was also applied on industrial effluents taken from textile and plastic factories at optimum studied conditions. The removal mechanism of metal ions followed

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adsorption and ion exchange processes. These results show that the MZ holds a great potential to remove heavy metals from industrial wastewater. The study recommends the reutilization of MZ as a potential adsorbent, which can greatly enhance the sustainability of useful resources.

Keywords: Pollutants; Adsorbents; Zeolite; Marble powder; Synthesis; Sustainability

1. Introduction

Increasing prices of raw materials and growing understanding of environmental issues have led to a change in the awareness of the value of waste materials. Waste recycling and reutilization are potentially encouraging routes toward growth of environment friendly, sustainable, and cost-effective industrial processes. As a result, it is of great interest to investigate the potential of turning wastes into valuable products. Conversion of marble powder into zeolitic-type materials has been shown to be a promising approach due to the similarity of the marble powder to some materials, precursors of natural zeolites. Hydrothermal and fusion treatment is the most widely used synthesis method for these materials [1,2]. The possibility of successfully producing zeolite from the waste marble powder is contributed to the fact that the marble powder has a considerable fraction of silica and alumina, which are the primary elements required for building the zeolite structure [3].

Zeolites are hydrated alumina silicates. Their structure consists of most important building blocks of inorganic tetrahedrons of silicon and alumina oxides. These atoms are bonded together with oxygen bridges to form well-defined channels and cavities. Zeolites are negatively charged as a result of the substitution of silica by alumina in the structure [4]. Water molecules and cations such as Na, K, and Ca are adsorbed onto the pore surfaces of zeolite. These cations equilibrium the negatively charged zeolite structure and are exchangeable species. These can be synthesized from different sources [5]. Two main groups have been classified for zeolites based on their Si/Al ratio and main applications. These groups include:

(1) high silica to alumina ratio with a large structure and used mainly as catalysts, for example, ZSM-5 zeolite; (2) medium to low silica to alumina ratio of fairly medium to small structures and used mainly as adsorbents and cation exchangers, for example, faujasite (X and Y types) and zeolite A [6,7].

The mechanism of adsorption by zeolite is ion exchange. In the three-dimensional structure of zeolite, there are large channels having negatively charged sites resulting from Al_3 replacement of Si_4 in the O_4 tetrahedral. The tetrahedral is linked by sharing oxygen atoms in rings and cages cavities occupied by cations. Zeolite mainly contains various types of cationic sites. The net negative charge of the anions is balanced by cations that occupy the channels within the structure, and can be replaced with heavy metal ions [8]. A wide variation in the cation exchange capacity of zeolite is due to a different nature of various structures of zeolite, adsorbed ions, natural structural defects, and their associated minerals. Structural defects, degree of hydration, variety of dimensions, and the presence of clays and other particles may cause differences in properties between zeolite [9].

The use of these materials as adsorbents has concerned much attention, given their large specific surface area, high adsorption capacity, and good cation exchange capability that assist to adsorb metals from aqueous media [10,11]. The discharge of water or pollution by textile and petroleum industries has become an important issue of environmental concern due to the significance of this natural resource in the energetic medium. In most cases, petroleum-related pollution is a chronic problem, due to activities and the outflow of urban and industrial wastewater contaminated with petroleum and its derivatives [12]. Industries discharge pollutants in liquid, solid, and gaseous form in the environment. Metal pollution is a problem linked with areas of severe industry. The term heavy metal refers to any metallic chemical element that has a relatively high density and is toxic or poisonous at low concentrations. The heavy metals include mercury (Hg), cadmium (Cd), arsenic (As), chromium (Cr), thallium (Tl), and lead (Pb) considered as toxic [13].

Heavy metals are dangerous because they have the ability to bioaccumulate. In order to remove these inorganic pollutants, various methods and technologies are used. Adsorption is one of the most practicable methods available due to its high removal efficiency for inorganic pollutants. Adsorption process may also provide an advantage over costly and energy concentrated alternative processes [14]. The adsorption process depends upon the type of adsorbent used [15].

This work aims to characterize the adsorption performance of zeolitic-type materials synthesized from marble powder waste by the treatment toward essential and non-essential elements using both synthetic solutions and contaminated industrial effluents. The intention is to assess the potential of converted waste powder as an adsorbent for wastewater treatment remediation applications, with a view of finding sustainable routes for the reutilization of the ashes.

2. Materials and methods

In this study, the marble powder was obtained from Cresent Marble Industries, Fazaia Road Rawalpindi, Pakistan.

The powdered waste marble is ground by mortar and pestle, then passed it through 40- μm sieve. Ten grams of waste marble mixed with 12 g of NaOH powder. It is ground to get homogeneous mixture. This homogeneous mixture is placed in crucible in furnace at 600˚C for 6 h. The resultant material is cooled at room temperature and ground to obtain the fused material. Ten grams of fused material is added in 10 mL of distilled water in conical flask. This is followed by aging process for 24 h by shaking on a shaker to obtain the aged material. Then, the aged material is heated in a sand bath at 80˚C for 12 h to obtain product. The aged material or product is filtered using whatman filter paper, washed it with distilled water, and placed in oven for overnight to obtain the synthesized product [16,17].

2.1. Characterization of synthesized product

The synthesized products were characterized by the properties listed as follows: (1) functional group identification, spectra of adsorbent recorded from 4,000 to 400 cm^{-1} on FTIR spectrophotometer (FTIR 8400, Shimadzu, Japan); (2) mineral species, by X-ray diffraction (XRD) analysis using Cu anode material, 45 kV, 40 mA, and a scanning rate of one degree per second from 5 to 79.9 or 80.0. The results of determined d-spacing and relative intensities for corresponding 2u values were analyzed by Xpert high score analytical Malaysia; (3) TGA; and (4) SEM of sample to identify the particle size. So doing, each of the mineral species including zeolite-like materials was identified according to the major and minor peaks. Due to the complexity of crystalline phases, the overlapping of peaks for different mineral species is a commonplace.

2.2. Batch adsorption studies

The laboratory-scale experimental studies were conducted using synthetic solutions prepared using deionized water with constant element concentration for each of Cd, Cr, Ni, Cu, Zn, and Pb separately. The uptake of heavy metals on the zeolite was carried out using the batch method. Batch adsorption experiments were conducted using 1 mg dose of zeolite with 10 mL of solution for each 10-min interval and these solutions containing heavy metal ions of desired concentrations (50, 100, 150, and 200 mg/L) for each metals at ambient conditions of room temperature and neutral pH. The solutions of six samples of each 10 mL were placed for 60 min. The applied time intervals were 10, 20, 30, 40, 50, and 60 min. After each interval of 10 min, the solutions containing heavy metals were filtered through whatman 40 filter paper using glass funnel and then the filtrate is checked on an ultraviolet light at desired wavelength.

3. Results and discussion

3.1. Characteristics of marble powder waste

After digestion of raw material, it was run on FAAS to check the metal concentration of Pb, Cd, Cr, Cu, Ni, and Zn. The metal concentrations of raw material are reported in Table 1.

3.2. Characteristics of synthesized product

The characterization of adsorbent material ZIA is done using FTIR and XRD analysis.

3.2.1. FTIR characterization of adsorbent

The FTIR characterization of adsorbent is shown in table. The major peaks and band were taken and studied to find out the functional group present in zeolite. Compare these spectrums to the standardized FTIR spectrum table. In zeolite, the following functional groups exist showed in Table 2 (FTIR 8400, Shimadzu, Japan).

Vibrations of the frameworks of zeolite in figure give rise to typical bands in the mid and far infrared.

Table 1
Metals concentration in marble powder waste

Metal	Concentration (mg/L)
Lead	7.2253
Cadmium	0.0646
Chromium	0.2955
Copper	0.7676
Nickel	2.5628
Zinc	3.9025

Table 2
FTIR characterization of adsorbents

Wave number (cm^{-1})	Assignment
3,600–3,650	Hydroxyl group
1,640	Adsorbed H_2O
1,438–1,452	Aluminum-containing entities; $Al_xO_y^{n+}$
1,250–920	Tetrahedral $SiO_{4/2}/AlO_{4/2}$
650–500	Si–O/Al–O bend; external linkages
200–50	Cationic vibration; far infrared region

A distinction is made between external and internal vibrations of the Si or Al $O_{4/2}$ tetrahedral [18]. The original assignments of the main IR bands in zeolite adsorbent ZIA are as follows: internal tetrahedral 1,250–920 cm^{-1}, asymmetrical stretch (n_{asym}); 720–650 cm^{-1}, symmetrical stretch (n_{sym}); 500–420 cm^{-1}, Si/Al–O bend; external linkages: 650–500 cm^{-1}, double-ring vibrations; 420–300 cm^{-1}, pore-opening vibrations; 1,150–1,050 cm^{-1}, asymmetrical stretch; and 820–750 cm^{-1}, symmetrical stretch. The positions of bands due to vibrations of external linkages are often very sensitive to structure [12,19].

Table 3
XRD identified pattern of compounds in hydrothermally treated zeolite

Visible	Ref. code	Score	Compound name	Displacement (°2Th.)	Scale factor	Chemical formula
*	01-086-2,335	48	Magnesium calcium carbonate	0.000	0.450	(Mg.064 Ca.936) (CO_3)
*	01-083-2,467	59	Silicon oxide	0.000	0.707	SiO_2
*	98-017-0,486	28	Zeolite	0.000	0.203	O_2Si_1
*	01-078-1,254	39	Silicon oxide	0.000	0.248	SiO_2

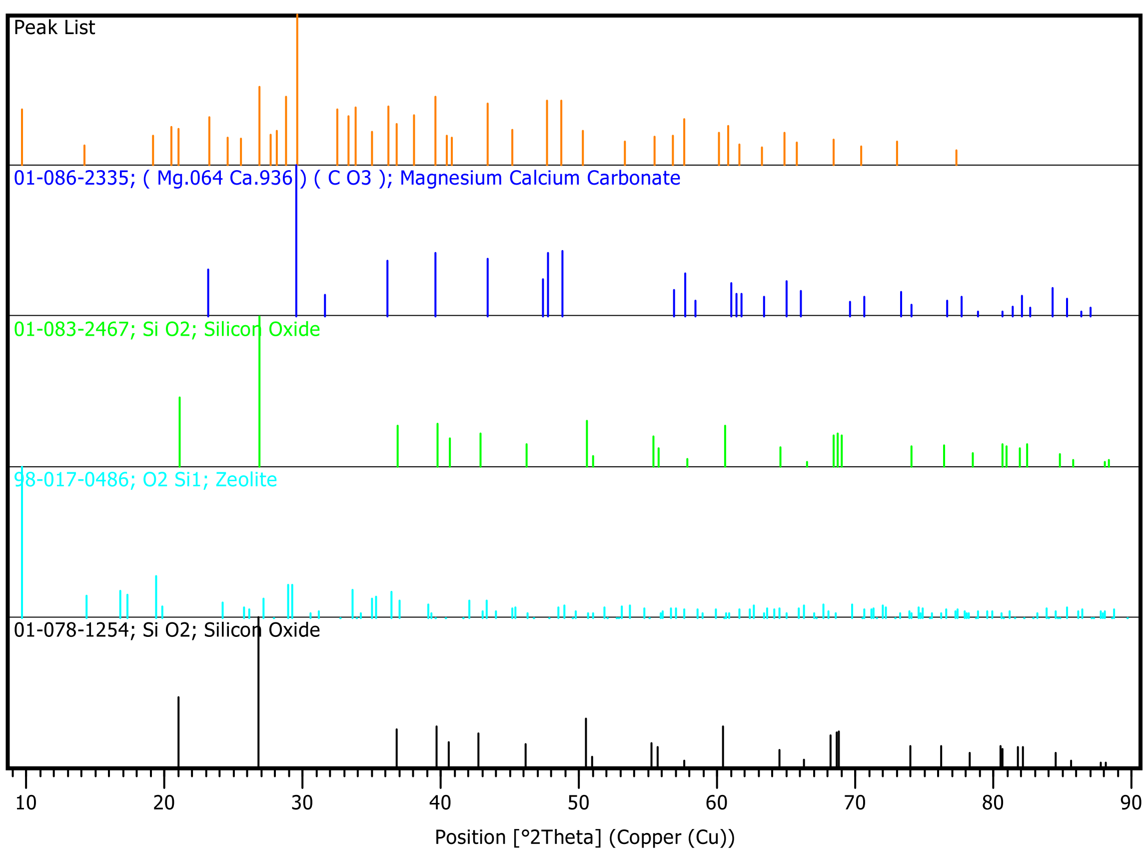

Fig. 1. Pattern of XRD of hydrothermally treated zeolite.

3.2.2. XRD analysis

Results of XRD analysis have proved that hydrothermal treatment processing is capable of converting marble powder into zeolite-like materials more efficiently as shown in Table 3. After synthesis, several types of zeolites have been identified on the basis of different conditions as given in Fig. 1. XRD powder pattern for the synthesized product resulted from various marble powder mineralize ratios is similar, but different in relative intensities for various mineral species [20,21].

3.2.3. TGA of synthesized product

The number of water molecules attached with the hydrothermally synthesized zeolite and its thermal stability were investigated using TGA. Upon heating the sample at room temperature, a continuous weight loss of 55% is clearly observed in TGA as given in Fig. 2. This weight loss is may be due to the dehydration of physically adsorbed water. When the sample is further heated in the temperature range of 200–700˚C, the weight loss observed is attributed to desorption of remaining water enclosed in the material matrix [22]. Reduced weight loss in this region was observed with an increase in crystallization temperature as well as Si/Al ratio of the sample which is consistent with the fact that zeolite becomes more hydrophobic as the Al content decreases [23,24].

3.2.4. SEM of synthesized product

The synthesized product is examined under an electronic microscope. The micrograph is presented in Fig. 3 using different magnifications. The sample revealed the presence of some cubic crystal of zeolite. The micrograph indicated a plate-like structure which is an indication that the silica and alumina are sliding over one another. Some partial damage of the plate-like structure was observed in the synthesized sample [25]. The SEM image revealed a uniform particle size of the sample with a regular shape. The synthesis SEM results give a narrow distribution of the particle size with an average crystal size of 1 μm [26].

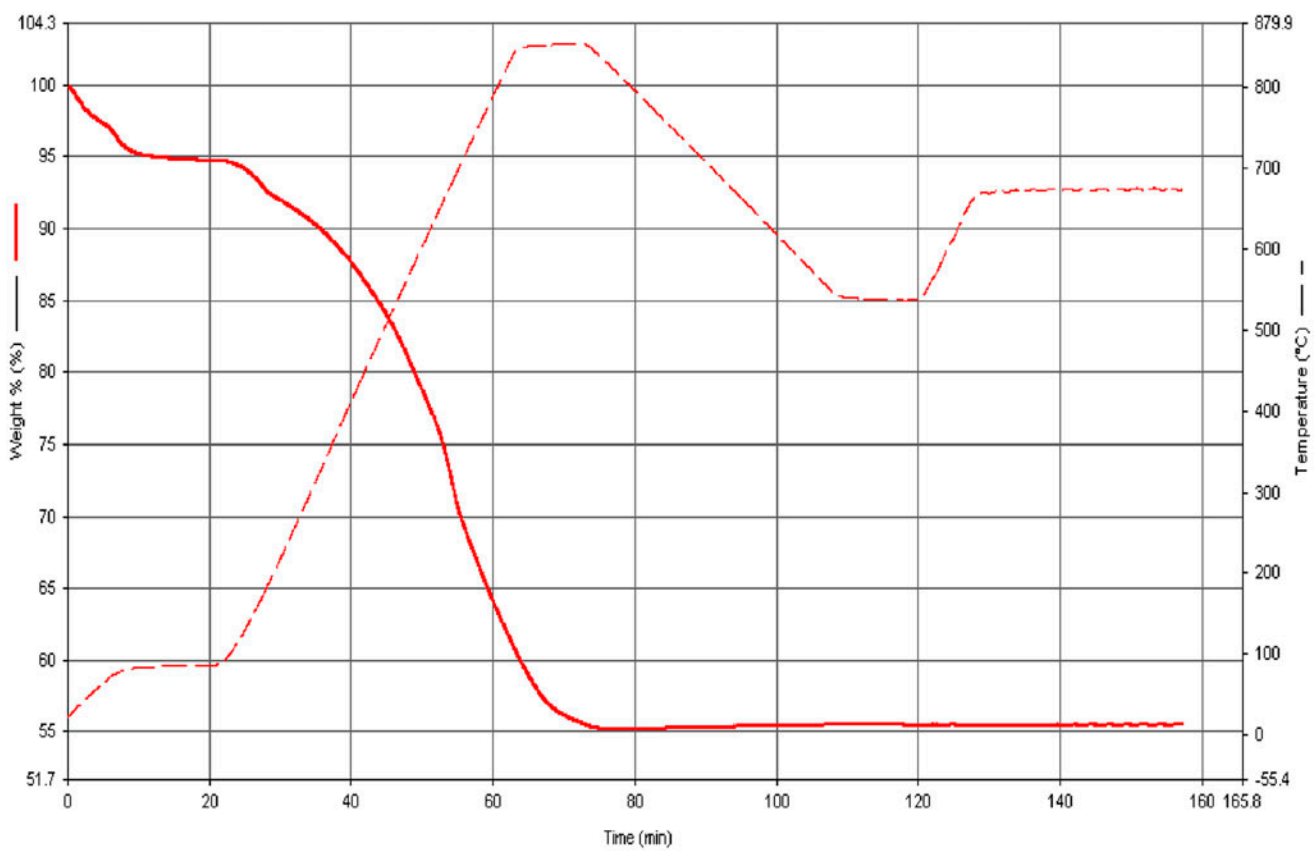

Fig. 2. TGA of synthesized adsorbent.

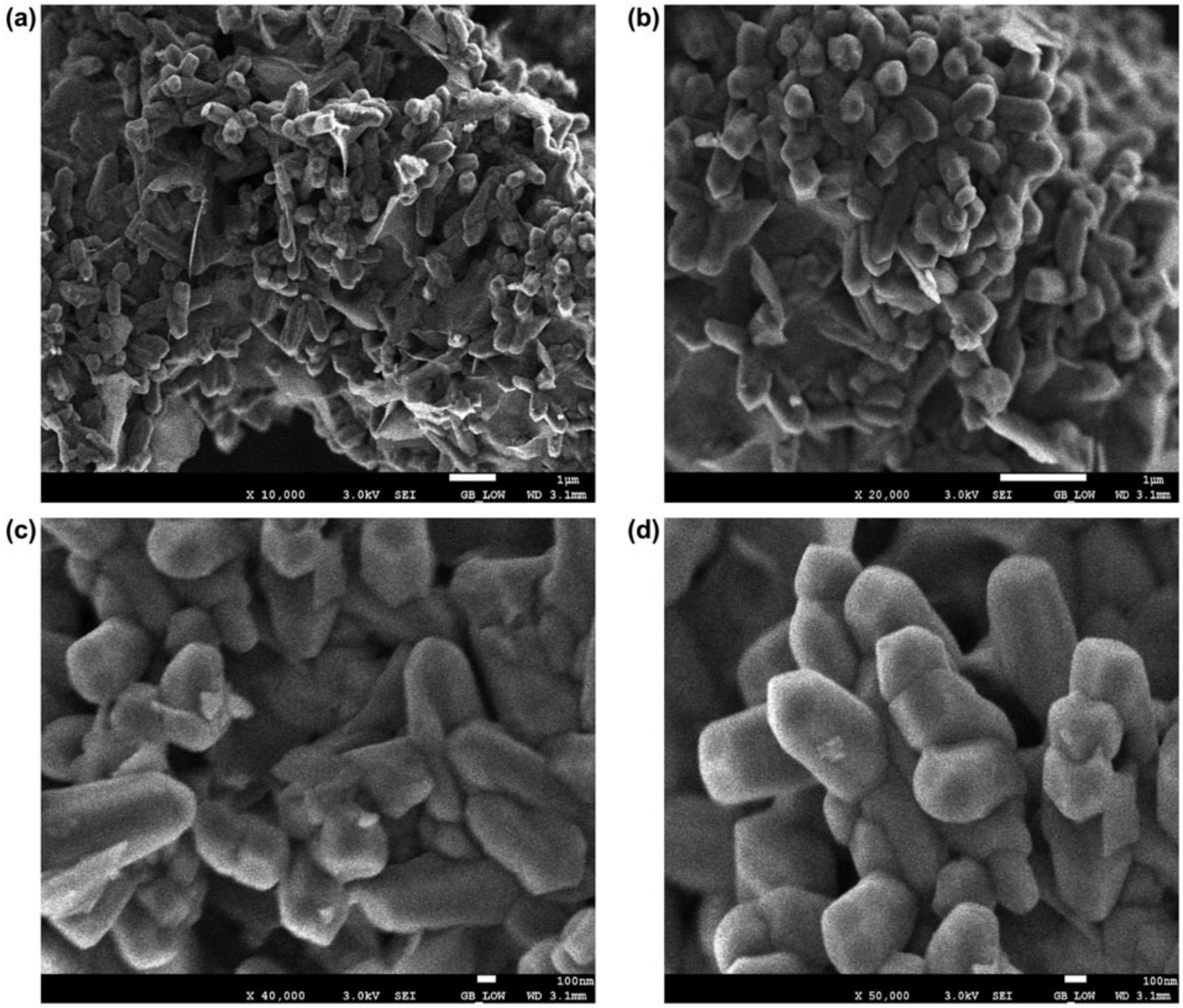

Fig. 3. SEM of adsorbent at different magnifications (a) 10,000×, (b) 20,000×, (c) 40,000×, and (d) 50,000×.

3.2.5. EDX of adsorbent

EDX graph of the adsorbent is shown in Fig. 4. The figure confirms the presence of C, O, Na, Al, K, and Si in the synthesized zeolite. Further studies in Table 4 show that the synthesized zeolite contains the following elements in it [15,27].

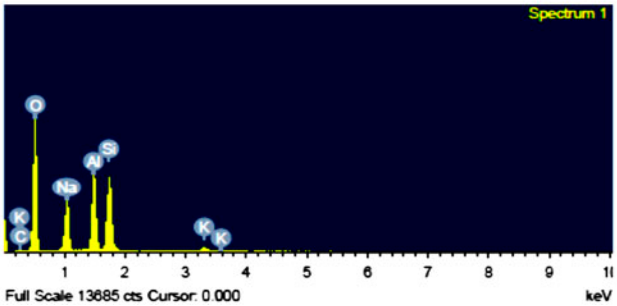

Fig. 4. EDX of adsorbent.

Table 4
Elemental composition of synthesized product

Element	Weight (%)	Atomic (%)
C	4.3	7.6
O	53.01	64.69
Na	10.77	9.00
Al	14.12	10.37
K	1.33	0.64
Si	17.12	10.21

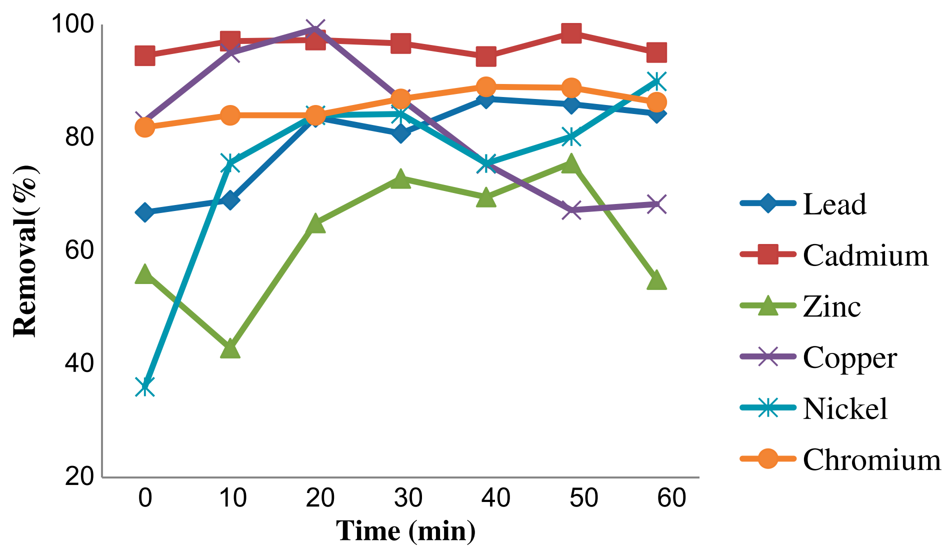

Fig. 5. Removal of metals using zeolite as an adsorbent at different contact times (optimum operating conditions).

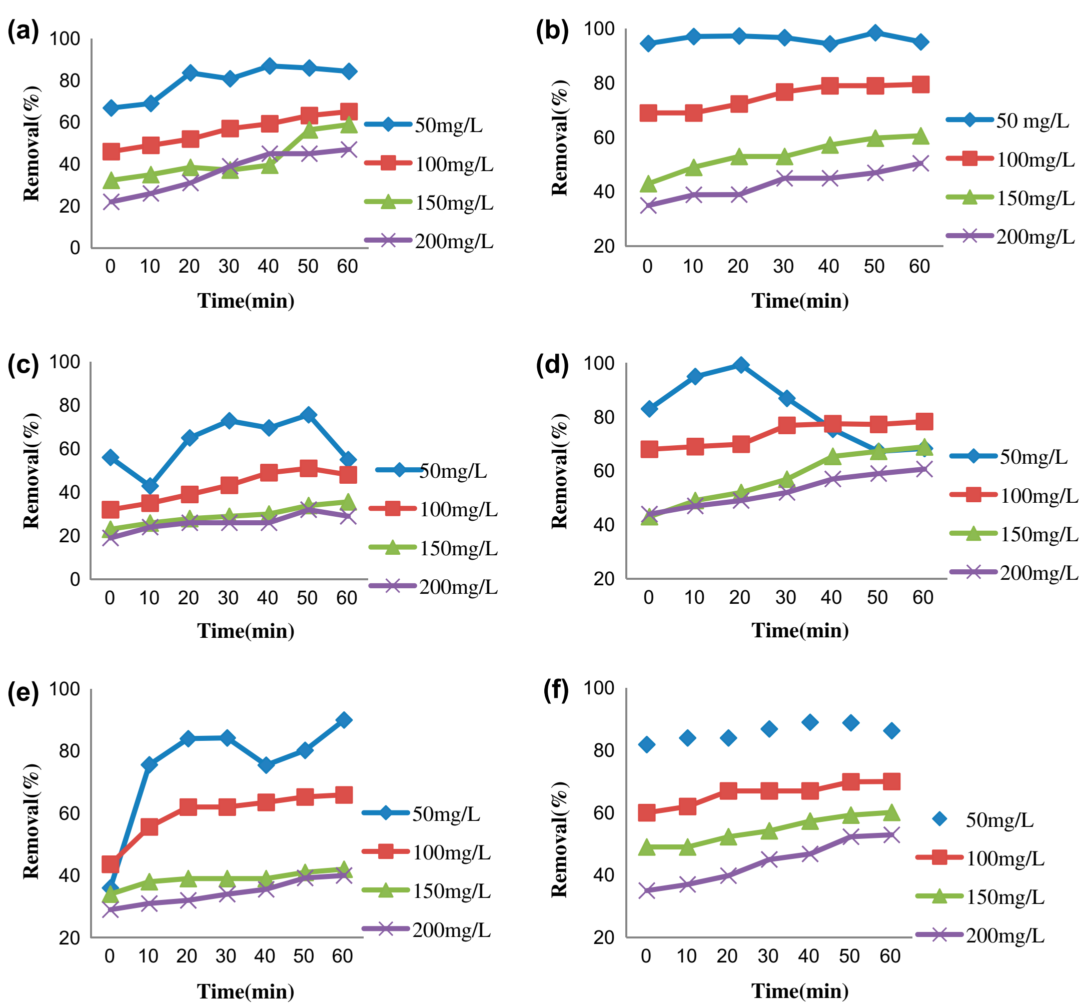

Fig. 6. Removal of metals (a) lead, (b) cadmium, (c) zinc, (d) copper, (e) nickel, and (f) chromium using M-Z adsorbent at different induced concentrations.

3.3. Adsorption of heavy metals

3.3.1. Effect of contact time

The studies of different contact times help in determine the removal of heavy metal from the constant mass of adsorbent (1 mg) at different time intervals of 10, 20, 30, 40, 50, and 60 min. The results given in Fig. 5 show that the adsorption of the studied heavy metals has a gradual increase with contact time because ions have maximum time to hold on to the surfaces of zeolite by physical or chemical bonding until 50 min, then the removal percent becomes constant, i.e. equilibrium is attained [19,28].

M-Z zeolite showed 90% removal of nickel at 50 mg/L and increases with time. The reason is that the raw material of M-Z zeolite contains high concentration of nickel and metal–metal binding occurs in it. M-Z showed maximum absorption of copper 99.29% and of cadmium 95.11, while CFA-Z and IA-Z zeolite showed 96.3 and 99.75% removal of cadmium at 50 mg/L, respectively. This may be attributed to the fact that both zeolites can act as ion exchange. Accordingly, the pore diameter is not effective, but the operating capacity depends upon other parameters such as concentration of adsorbent, contact time, and particle size [28,29].

3.3.2. Effect of induced concentrations

Four different concentrations (50, 100, 150, and 200 mg/L) of heavy metals solution were introduced on zeolite adsorbent with different contact times. It is investigated from data that the change in induced concentration has a significant impact on adsorption process. It is deducted that adsorbent showed the decreasing trend of adsorption with an increase in induced concentrations of metal solutions [18,29]. The reason behind decrease in adsorption is attributed to the less number of available adsorption active sites as compared to heavy metal concentrations [27].

From the result showed in Fig. 6, the percentage removal at initial concentrations of the heavy metals are high, particularly for lead, cadmium, and chromium, whereby the ability to absorb Pb, Cd, and Cr cations at higher concentrations is less because it involved energetically less favorable sites of zeolite in the uptake of heavy metals. The higher metal uptake at low concentration is due to the availability of greater surface area with active centers on the adsorbent for lesser amounts of adsorbate ions [27].

3.3.3. Removal of metals

The adsorption performance of M-Z was generally superior to that of the natural zeolite for both synthetic heavy metal solutions and contaminated industrial effluents. Zeolite achieved the removal of cadmium, chromium, lead, copper, nickel, and zinc as given in Table 5 from 1 mg sorbent dosage [30].

3.4. Application on industrial effluents

The industrial effluents taken from plastic and textile factories from Faisalabad as shown in Tables 5 and 6 were subjected to batch removal for the removal of metals using zeolite synthesized from marble waste under varying experimental parameters. The results were analyzed on FAAS. The 1 mg dose of zeolite was placed in each of 10-mL effluent sample taken from different industries. The effluents were placed for 60 min at room temperature and neutral pH for the removal of metals. The results are shown in Fig. 7, Table 7 and it is

Table 5
Removal of metals by MZ

Metals	Removal percentage (%)
Lead	86.90
Cadmium	98.50
Zinc	75.60
Copper	99.29
Nickel	84.20
Chromium	89.04

Table 6
Physiochemical parameters in industrial effluents

Physiochemical parameters	Plastic industry effluent	Textile industry effluent
pH	9.37	8.92
EC (ms/cm)	6.38	9.08
SO_4^{-2} (mg/L)	500	550
Cl^{-1} (mg/L)	650	720
COD (mg/L)	160	190

Table 7
Metals composition in industrial effluents

Concentration of metals	Plastic industry effluent (mg/L)	Textile industry effluent (mg/L)
Lead	3.0421	3.4268
Cadmium	2.1103	1.0013
Chromium	1.0028	1.9004
Copper	2.0028	2.9004
Nickel	1.2567	2.0978
Zinc	1.425	1.936

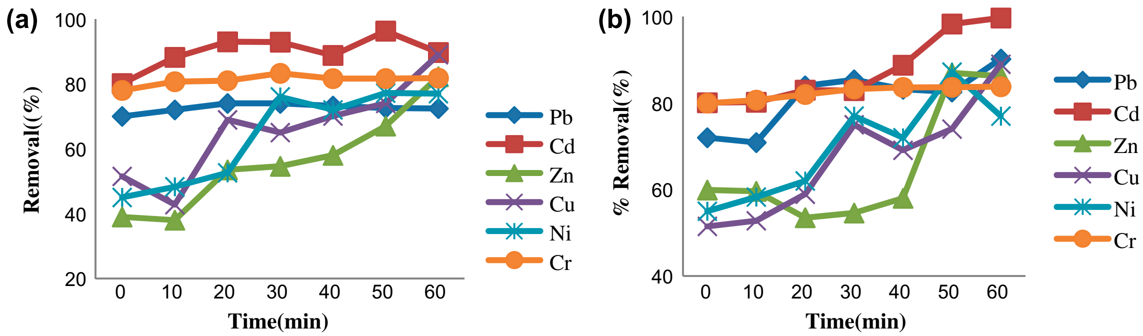

Fig. 7. Removal of metals using M-Z adsorbent from industrial effluents (a) plastic and (b) textile factories.

investigated that M-Z is the best adsorbent and its removal efficiency is 99.99% in industrial effluents which is quite useful for the removal of metals [31].

4. Conclusion

The conclusions of the present research work show that the attempt to produce zeolite materials from marble powder waste. It seems that introducing a fusion step is an essential step to ensure the dissolution of the marble particles and to ensure the formation of materials that are necessary for synthesis of zeolite. The zeolitic-type materials synthesized by marble powder demonstrated the promising adsorption performance for the elements tested (Cd^{2+}, Cr^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, and Pb^{2+}).

The key findings of this study are:

(1) Adsorption studies indicated higher affinity for Cd, Cr, Ni, Cu, Zn, and Pb for zeolite.
(2) The sequence of average removal of metals at optimum operating conditions on M-Z as:

Cu > Cd > Cr > Pb > Ni > Zn

Given the positive results found in this study, further work aims to optimize the performance of the marble zeolite (MZ). The affinity of this material for heavy metals, its flexibility, and low-cost also opens the possibility for its application in other industrial processes, including chemical and catalyst synthesis. In any case, the reutilization of incinerator ash achieves the goal of reducing the burden of industry on its safe disposal and improves the sustainability of waste incineration technology.

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