



## New method for the adsorption of organic pollutants using natural zeolite incinerator ash (ZIA) and its application as an environmentally friendly and cost-effective adsorbent

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### ABSTRACT

Rapid development of civilization together with the evolution of industry has caused deep changes in the quality of the environment in which the human beings live. The enhancing demands for a healthy environment, particularly for the removal of pollutants from industrial and municipal wastewater, are a major reason to search for new materials. Zeolites are potential materials and can conveniently be used as adsorbents for the removal of environmental pollutants. The present research offers a green approach to investigate the removal efficiency of polyaromatic hydrocarbons and azo dyes from wastewater using zeolite derived from incinerator waste (ash) as an effective, economical, and environmentally friendly suitable material as an adsorbent. The significance rests on the use of hazardous waste into a useful resource (adsorbent). Batch adsorption studies are conducted and results are analyzed by UV–vis spectrophotometer. The results depicted the performance of zeolite incinerator ash (1 mg/kg) in removing 98, 97, and 96% of anthracene, phenanthrene, and pyrene, respectively, and up to 90% removal of the dyes upon adsorbate–adsorbent contact for 10 min. Thus, the zeolite made from natural waste materials possess unique porous properties and offer promise for the removal of organic pollutants. The recovery ability can also be tested and evaluated to determine the reusability of the adsorbent. This is an additional benefit which reduces the need for extracting materials from the environment to be used in the industrial processes.

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## 1. Introduction

The discharge of wastewater from industries has become a significant issue of environmental concern due to importance of this natural resource in the energetic matrix. These industries release organic pollutants that include pesticides, fertilizers, hydrocarbons, phenols, plasticizers, biphenyls, dyes, and oils. In most cases, petroleum related pollution is a chronic problem due to port activities and the outflow of urban and industrial wastewater contaminated with petroleum and its derivatives [1]. Polycyclic aromatic hydrocarbons (PAHs) are one of the major groups of these organic contaminants. They are persistent semi-volatile contaminants that can remain in the environment for long periods due to their high degree of conjugation and aromaticity [2]. Many industries also produce highly colored wastewater. Dyes are considered as type of organic pollutants. The textile, pulp, and paper industries are reported to utilize large quantities of a number of dyes; these pollutants may be found in wastewaters of many industries generating considerable amounts of colored wastewaters, toxic, and even carcinogenic, posing serious hazard to living organisms [3]. Azo dyes are the most difficult to treat constituent of the textile wastewater. Reactive dyes are difficult to biodegrade in an environment. Hence, their presence in wastewater is undesirable, and it is essential to remove coloring material from effluents before being discharged in the environment. This is important in those regions, where water resources are limited [4]. The conventional methods used for removal of these pollutants have some drawbacks. They simply move the contamination elsewhere and may create significant risks in the excavation and treatment of hazardous material. In order to treat these organic pollutants, various methods and technologies are used. Adsorption offers a good solution for cleaning many waste streams by adsorbing industrial effluents such as dyes and PAHs. A major advantage of adsorption lies in the fact that the persistent compounds are removed, rather than being broken down to potentially dangerous metabolites that may be produced by oxidation and reductive processes [5]. The adsorption process depends upon the type of adsorbent used and the economic capability of adsorptive processes is largely due to the versatility of adsorbent used.

Large amount of waste that is generated from the municipal activities can be converted to useful

products [6]. The present environmental concerns over municipal solid waste ash disposal have sparked a resurgent interest in its conversion to value added product such as zeolites. Municipal solid waste ash has a tremendous potential for conversion to zeolite and its subsequent utilization as adsorbents for the removal of organic pollutants in the industry [7]. Although variety of different adsorbents is available, but the low cost of natural zeolites is often cited as a major incentive for their use. Zeolites belong to a group of hydrated aluminosilicates of the alkali or alkaline earth metals. Natural zeolites are environmentally and economically acceptable hydrated aluminosilicate materials with exceptional ion exchange and sorption properties. The unique porous structure gives natural zeolites various application possibilities [8]. It is a remarkable natural mineral because of its extraordinary ability to absorb, hold, release, and exchange different chemicals, nutrients, toxins, and ions according to need. In general, they contain silicon, aluminum, and oxygen in their framework and cations, water, and other molecules within their pores [9]. The efficiency of water treatment using natural zeolites depends on the type and quantity of the used zeolite, the size distribution of zeolite particles, the initial concentration of contaminants (cation/anion), pH value of solution, ionic strength of solution, temperature, pressure, contact time of system zeolite/solution, and the presence of other organic compounds and anions. Recycle tests showed that the zeolite could be cleaned and reused at least three times without significant reduction in treatment effectiveness [10].

Thus, the goal of this study is to investigate the removal ability of zeolite incinerator ash (ZIA) for harmful organic compounds from wastewater and to find original ways to utilize zeolite incinerator ash as an adsorbent that is environmentally friendly and novel, as it eliminates the use of costly adsorbents and shows the trend toward green chemistry where waste materials are competently utilized.

## 2. Materials and methods

The laboratory scale experimental studies were conducted by synthetic solutions prepared using ethanol for PAHs and deionized water for dyes with constant concentration for each of them separately. The uptake of PAHs and the dyes on the zeolite incinerator ash was carried out using the batch method.

The adsorption technique is used for the removal of three selected Polyaromatic hydrocarbons (phenanthrene, anthracene, and pyrene) and dyes (Eriochrome black T and Methyl orange).

### 2.1. Preparation of adsorbent

The adsorbent zeolite incinerator ash was taken from synthetic standardized protocol [11].

### 2.2. Characterization of adsorbent

The material synthesized from standard protocol was taken and it was characterized by the properties listed as follows:

(1) Functional group identification, it was mixed with KBr in 1:10 and pressed into pellet. Spectra of adsorbent recorded from 4,000 to 400  $\text{cm}^{-1}$  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 808 for  $2\theta$ . The results of determined d-spacing and relative intensities for corresponding  $2\theta$  values were analyzed by X'pert High score PANalytical Malaysia. (3) SEM of sample to identify particle size. In 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.

### 2.3. Batch adsorption studies

For PAHs, the solid/volume ratio of 1 mg per 5 ml was used. The batch adsorption studies were carried out by varying initial concentration of solution (0.01 and 0.02 mg/L), and then adding a known volume (5 ml) of prepared solution into a number of glass vials containing a known amount of adsorbent (1 mg) at ambient conditions. The solutions were placed for 10 min and after that solutions were filtered through Whatman 40 filter paper. Each batch experiment applied a virgin adsorbent using an open batch. The aliquot was drawn after regular time intervals till equilibrium was attained between adsorbate and adsorbent. While for azo dyes; the solid/volume ratio of 1 mg per 5 ml was used. Eriochrome black T and Methyl orange were studied at parameters of initial concentration (0.001, 0.01, and 0.1 mg/L), mass of adsorbent (1 mg), and contact time (0, 10, 20, 30, 40, 50, and 60 min) at ambient conditions.

After filtration, PAHs and dyes were quantified by UV-visible Spectrophotometer (UV-1601, Shimadzu,

Japan) and absorbance of unabsorbed sample was recorded. The adsorbed concentration was determined from the standard calibration curves.

Percentage removal was calculated using the formula:

$$\frac{C_i - C_t}{C_i} \times 100$$

whereas;  $C_i$  is Initial concentration,  $C_t$  is Final concentration.

Absorbance of phenanthrene, anthracene, and pyrene was recorded at 250, 260, and 241 nm, respectively, and the maximum wavelengths for azo dyes on UV-vis spectrometer were 220 and 464 for Eriochrome black T and Methyl orange, respectively.

## 3. Results and discussion

### 3.1. Characterization of adsorbent material

The characterization of adsorbent material ZIA is done by doing following analysis.

#### 3.1.1. FTIR characterization of adsorbent

The FTIR characterization of the adsorbent is shown in Fig. 1. In it, the major peaks and bands were taken and studied to find out the functional groups present in zeolite. Then these spectrums were compared to the standardized FTIR spectrum table. In all types of zeolite the following functional groups exist as shown in Table 1 (FTIR 8400, Shimadzu, Japan).

The FTIR spectra of zeolite adsorbent are in the range of 4,000 to 400  $\text{cm}^{-1}$ . The absorption features are resulted from stretching and bending modes of the Si or Al in zeolite framework. Vibrations of the frameworks of zeolites in following figure give rise to typical bands in the mid and far infrared. A distinction is made between external and internal vibrations of the Si or Al  $\text{O}_4/2$  tetrahedral. The original assignments of the main IR bands in zeolite adsorbent ZIA is as follows: internal tetrahedral: 1,250–920  $\text{cm}^{-1}$ , asymmetrical stretch ( $n_{\text{asym}}$ ); 720–650  $\text{cm}^{-1}$ , symmetrical stretch ( $n_{\text{sym}}$ ); 500–420  $\text{cm}^{-1}$ , Si/Al–O bend; external linkages: 650–500  $\text{cm}^{-1}$ , double ring vibrations; 420–300  $\text{cm}^{-1}$ , pore opening vibrations; 1,150–1,050  $\text{cm}^{-1}$ , asymmetrical stretch; 820–750  $\text{cm}^{-1}$ , symmetrical stretch. The positions of bands due to vibrations of external linkages are often very sensitive to structure [12].

Zeolitic vibrational spectra are usually very complicated, because in addition to the framework structure, the spectra are also influenced by other factors such as the existence and the nature of charge

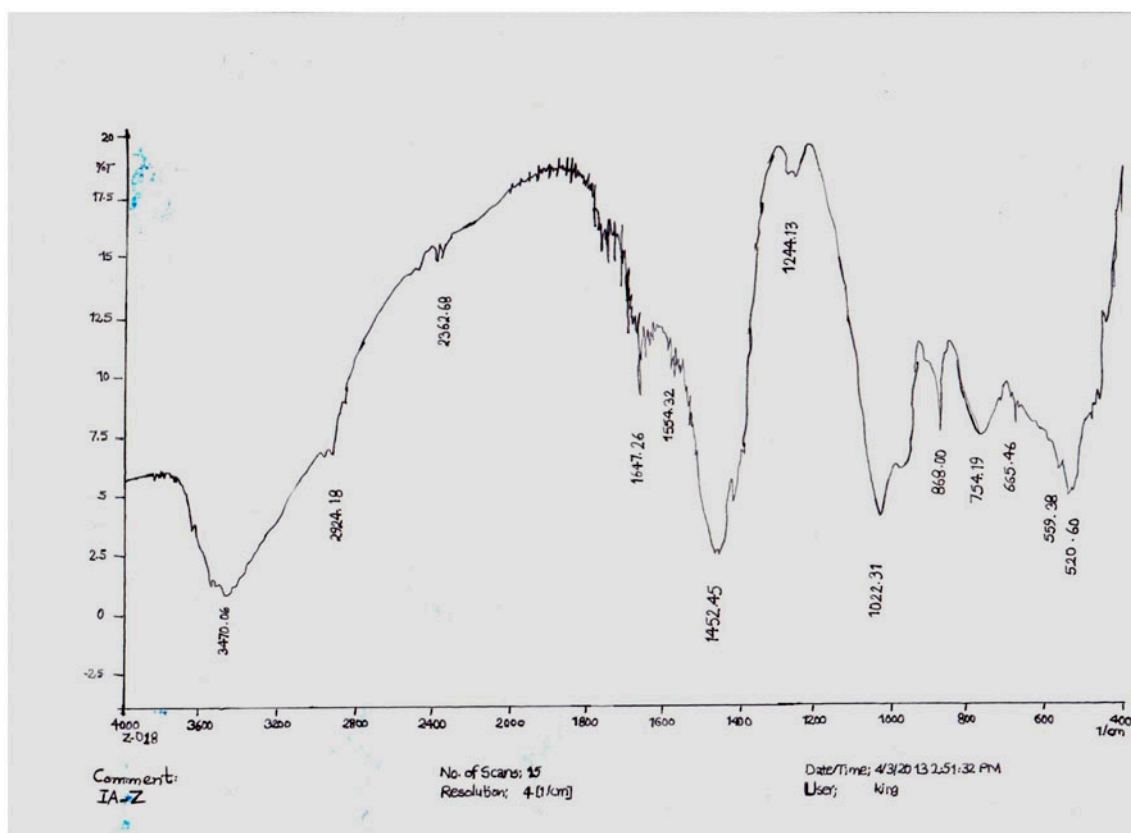


Fig. 1. FTIR characterization of adsorbent ZIA.

Table 1  
FTIR Characterization of adsorbent

Wave number ( $\text{cm}^{-1}$ )	Assignment
3,600–3,650	Hydroxyl group
1,640	Adsorbed $\text{H}_2\text{O}$
1,438–1,452	Aluminum containing entities; $\text{Al}_x\text{O}_y^{n+}$
1,250–920	Tetrahedral $\text{SiO}_4/2/ \text{AlO}_4/2$
650–500	Si–O/Al–O bend; external linkages
200–50	Cationic vibration; far infrared region

balancing cations, the degree of hydration, and the Si/Al ratio. A study by Li and Wu, investigated in detail the vibrational spectra of natural zeolites belonging to six structural groups [13]. This study also proves that vibrational spectra are useful for the identification of zeolite structures.

### 3.1.2. XRD analysis

XRD powder pattern for the adsorbent ZIA resulted from various IA mineralizer ratios are similar, but different in relative intensities for various mineral

species [14]; whereas, magnesium calcium carbonate and silicon oxide present in adsorbent showed the existence of zeolite shown in Fig. 2 [15] (Table 2).

### 3.1.3. SEM of adsorbent

The adsorbent (ZIA) was examined under an electronic microscope. Using different magnifications, the micrograph is presented in Fig. 3. The sample showed 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

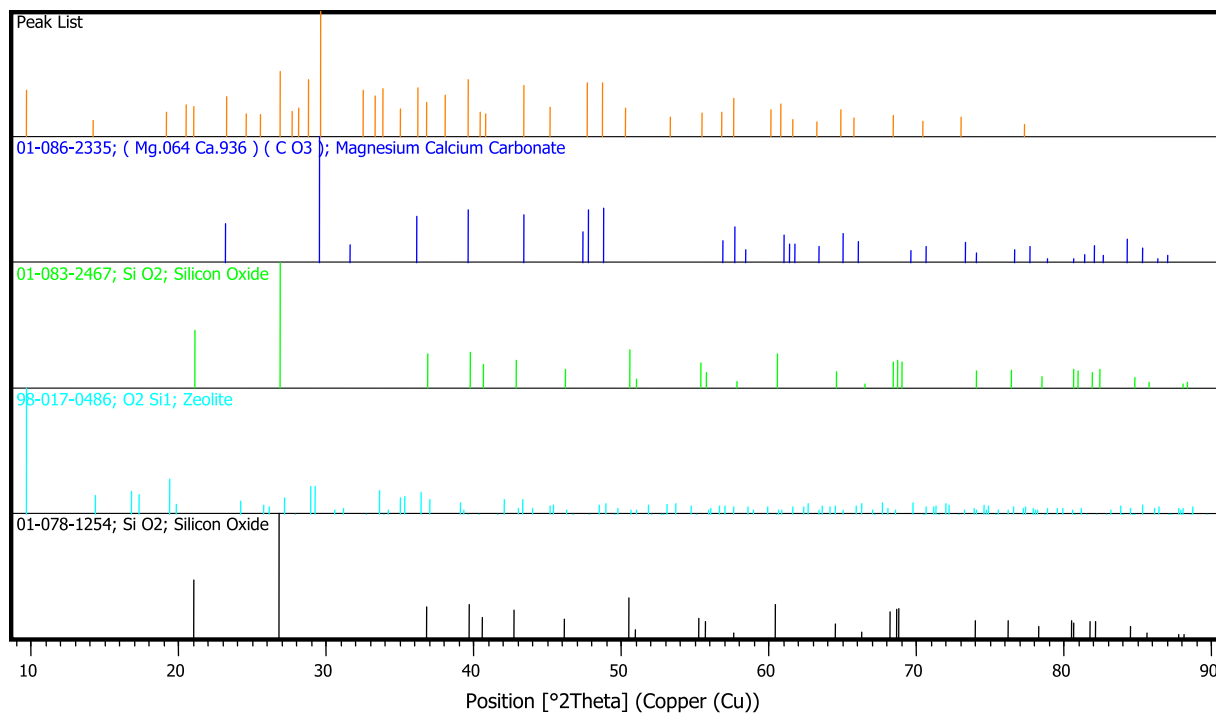


Fig. 2. Pattern of XRD of adsorbent.

Table 2  
XRD identified pattern of compounds in adsorbent (ZIA)

Visible	Ref. code	Score	Compound name	Displacement [°2Th.]	Scale factor	Chemical formula
*	01-086-2335	58	Magnesium calcium carbonate	0.000	0.820	(Mg.064 Ca.936) (CO <sub>3</sub> )
*	01-083-2467	39	Silicon oxide	0.000	0.277	SiO <sub>2</sub>
*	98-017-0486	27	Zeolite	0.000	0.133	O <sub>2</sub> Si <sub>1</sub>
*	01-078-1254	41	Silicon oxide	0.000	0.238	SiO <sub>2</sub>

over one another. Some partial damage of the plate-like structure was observed in the synthesized sample. The SEM image revealed a uniform particle size of the sample with a regular shape. The synthesis SEM results give a narrow distribution of particle size with average crystal size of 1  $\mu\text{m}$  [15].

### 3.2. Batch adsorption experiments

Batch adsorption experiments were conducted to determine the removal efficiency of selected waste material as adsorbent. Parameters like induced concentration and contact time are studied to check the level of adsorption. For each batch experiment, virgin adsorbent was used for the removal of organic pollutants. Three PAHs were selected for the present investigation that includes phenanthrene, anthracene,

and pyrene and they are symbolically represented as Phe, Ant, and Pyr, respectively. While two dyes were selected that includes Eriochrome black T and Methyl orange.

#### 3.2.1. Adsorption of PAHs

The first parameter studied was the effect of contact time on the adsorption of PAHs. As equilibrium time is one of the most important parameter in determining the cost-effective behavior and efficiency of adsorbents. The studies of different contact time helps in determining the removal of PAHs from constant mass of adsorbent (1 mg) at different time intervals of 5, 10, 15, 20, 25, and 30 min. The percentage removal of the adsorbent was found to be increased with contact time and it becomes constant with increase in



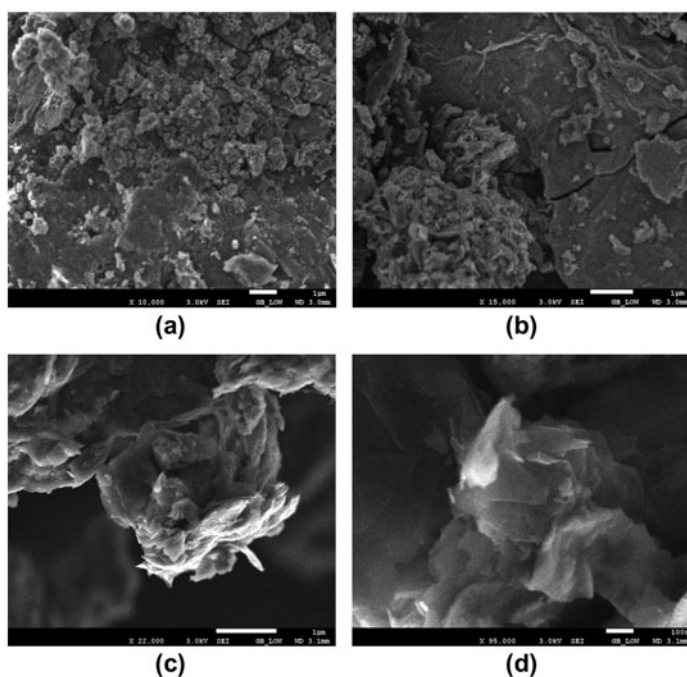


Fig. 3. SEM of adsorbent at different magnifications (a) 10,000X, (b) 15,000X, (c) 22,000X, and (d) 95,000X.

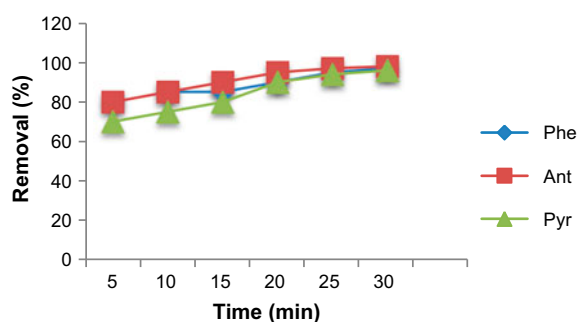


Fig. 4. Removal of PAHs using ZIA as an adsorbent at different contact time (optimum operating conditions).

contact time. As adsorption of PAHs is a dynamic equilibrium, in some cases more than one equilibrium times are established as given in Fig. 4.

From the calculated removal percentage, the efficiency of ZIA is 98, 97, and 96% of anthracene, phenanthrene, and pyrene, respectively, as shown in Fig. 5. The rapid adsorption of PAHs by zeolites is attributed to the external surface adsorption. Since all the adsorbent sites exist in the exterior of the adsorbent, it is easy for the adsorbate to access these active sites, resulting in rapid approach of equilibrium [16]. Adsorption equilibrium is attained approximately within 10–15 min with zeolite. However, their different

adsorption capacities depend on their molecular size and volatility.

The second parameter studied was the effect of induced concentrations for the adsorption of PAHs. Two different concentrations (0.01 and 0.02 mg/L) were introduced on ZIA adsorbent. It is noticed from the data that change in concentration is comparable and there is no significant difference in the results. But the 0.02 mg/L of concentration gives better percentage removal, which indicates that the higher the concentration, the higher is the percentage removal. This trend shows that the removal process was faster at higher concentrations for smaller PAH's that have lower molecular weights such as phenanthrene which suggests a diffusive mass transfer controlled process. For diffusion controlled process, the rate of uptake should increase with the concentration gradient, the driving force for diffusion, and therefore increase with the initial concentration. The adsorption capacity of the ZIA used was greater for phenanthrene and anthracene as compared to pyrene, as they have a smaller size which makes it easier for its molecules to diffuse into more micro pores for adsorption process [17]. This further gives proof that there is a form of molecular sieving in the adsorption process with some portion of the micro pores being inaccessible to some PAH molecules [18,19].

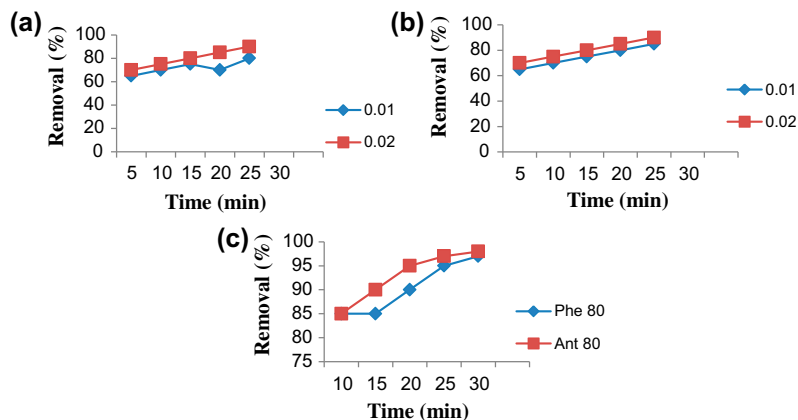


Fig. 5. Removal of PAHs (a) Phe, (b) Ant, and (c) Pyr using ZIA adsorbent at different induced concentrations.

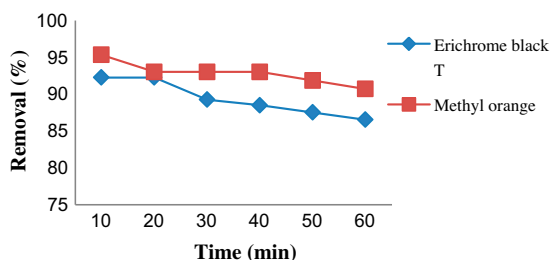


Fig. 6. Removal of azo dyes using adsorbent ZIA at different contact time (at optimum operating conditions).

### 3.2.2. Adsorption of dyes

The synthetic solution of Eriochrome black T and Methyl orange were subjected to batch removal using ZIA. The stock solution of dyes with the concentration of 10 mg/L was prepared and dilutions of 0.001, 0.01, and 0.1 mg/L were made with distilled water. The 1 mg dose of zeolite was placed in each solution of different concentrations. The solutions were placed for 60 min. The samples were then filtered and analyzed by UV Spectrophotometer [20].

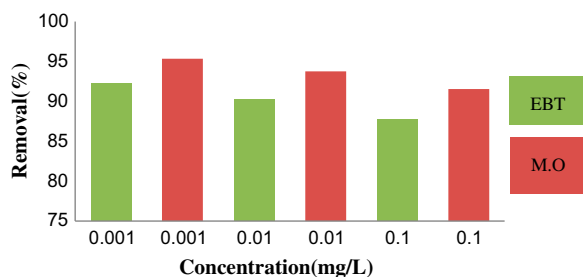


Fig. 7. Removal of azo dyes using adsorbent ZIA at different induced concentration.

Eriochrome black T is a complexometric indicator, while Methyl orange is a universal indicator and both have sodium ion in it. It forms a complex with calcium, magnesium, or other metal ions [21].

Firstly, the effect of contact time on the amount of azo dyes adsorbed was investigated at 1 mg mass of adsorbent and optimum initial concentration. The time dependent behavior of dye adsorption was examined by varying the contact time between adsorbate and adsorbent in the range of 5–30 min [22]. The removal of Eriochrome black T and Methyl orange dyes by adsorption increased with time and attained a maximum value of 90% in 10 min and thereafter it remained constant for all the concentrations studied [23]. It can be observed in Fig. 6.

It is investigated that the natural zeolite usually has some ion exchangeable cations such as Na, K, Ca, and Mg in channels. Such cations give a high cation exchange capability to zeolite, hence it can be exchanged with organic such as cationic dyes like Methyl orange [24].

The second parameter studied was the effect of induced concentrations for the adsorption of dyes given in Fig. 7. Three different concentrations (0.001, 0.01, and 0.1 mg/L) of azo dyes solution were introduced on ZIA adsorbent with different contact time [25]. It indicated that the ZIA adsorbent showed the decreasing trend of adsorption with increase in induced concentrations at 1 mg dose of adsorbent. The reason behind decrease in adsorption is attributed to the less number of available adsorption active sites as compared to azo dyes concentrations [26]. The adsorption percentage removal for dyes is 97.7 and 92.27% at 0.001 mg/L and 89.9 and 86.5% at 0.1 mg/L for Eriochrome black T. While for Methyl orange, the results showed the percentage

removal of 98.8 and 93.4% at 0.001 mg/L and 85.8 and 90.25% at 0.1 mg/L using ZIA adsorbent [27].

#### 4. Conclusion

The key findings of this study include:

- (1) The adsorption performance of zeolite incinerator ash was generally superior to that of the natural zeolite for both PAHs and azo dyes. ZIA achieved 98, 97, and 96% of anthracene, phenanthrene, and pyrene, respectively, and for dyes 90% in 10 min from 1 mg adsorbent dosage.
- (2) The sequence of average removal of PAHs at optimum operating conditions on ZIA as Ant > Phe > Pyr.
- (3) The sequence of average removal of dyes at optimum operating conditions on ZIA as Methyl orange > Eriochrome black T.
- (4) The application of ZIA for the remediation of organic pollutants is a cost-effective, simple, and environmentally save means of wastewater treatment. This study shows that there is a potential to produce zeolite from waste material such as incinerator ash.
- (5) The affinity of the adsorbent for organic pollutants, in addition to their flexibility and low cost, also allows the possibility of its application in other industrial processes. Furthermore, the reutilization of waste achieves the goal of reducing the burden of industry to practice safe disposal and improves the sustainability of waste incineration technologies.

Given the positive results found in this study, further work aims to optimize the performance of the ZIA. The affinity of this material for organic pollutants, its flexibility and low cost, also open 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. Thus, this study shows the trend toward green chemistry where naturally synthesized materials are efficiently utilized.

#### Conflict of Interest

The authors certify that there is no conflict of interest with any financial organization regarding the material discussed in the paper.

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