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RESEARCH ARTICLE

SYNTHESIS AND CHARACTERIZATION OF ZEOLITE LINDE W AND ITS APPLICATION AS ADSORBENT FOR MALATHION

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ABSTRACT

Hydrothermal synthesis was carried out for synthesis of Zeolite W under the static conditions. The synthesize sample was characterized by X-ray diffraction (XRD), Scanning Electron Microscope-Energy Dispersive spectroscopy (SEM–EDAX) and Fourier Transform Infra Red (FTIR) spectrocopy. Zeolite W was used as an adsorbent for the adsorption of malathion from aqueous solution. Adsorption studies were performed in a batch system. The effects of various experimental parameters (contact time, solution pH, effect of temperature and adsorbent doses) on the adsorption efficiency of zeolite W against were investigated for malathion. Zeolite W also performed against malathion in acidic and basic condition but highest adsorption capacity observed at pH 7 (aqueous solution).

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INTRODUCTION

Generally, in agriculture production of crops depends upon land, water, fertilizers, seeds and pesticides. Pesticides are an essential part of crop growing. It is chemical method which is used in crop for controlling insects, diseases and weeds in multiple crops and it is well-known effective technology for growing crops. Due to their toxic effect in nature and health hazard for human beings, they are very hazardous and unsafe in nature (Gupta, 2002). The pesticides such as herbicides, insecticides, fungicides and rodenticides, etc. enter in the atmosphere through farming, household and industrialized activities (Martínez, 2000 and Hernández-Soriano, 2007). According to above demand of now days, it is required to reduce and eliminate these life threatening compounds effect from wastewater and environment before releasing their toxic effect in the environment. It is proved that surface adsorption procedure is an excellent method for treating effluents. It is also an advantage and conventional processes, towards environment (Ravikumar, 2007). Adsorption is low cost, simple design, availability and ability to treat wastes in more concentrated form. Therefore, it is considered to be relatively better to other techniques (Kannan 2001 and Meshko, 2001). Toxins are moved from an aqueous medium onto the surface of the solid material in this process. Adsorbents with high surface reactivity, surface area and with a high number of the vacant site are in demand. Several natural or synthetic adsorbents like polymers, activated carbon, agro-industrial wastes, zeolite and CNTs (Ai, 2011; Ansari, 2010 and Hasan, 2008) have been used as adsorbents for the treatment of contaminated water. Natural and synthetic zeolites are widely used in many applications related to field of agriculture due to their unique physicochemical properties. They are being highly used in the production of mineral fertilizers with slow release and as carriers of active ingredients of herbicides, fungicides and pesticides (Szerement, 2014). Zeolites are a group of tectosilicates of ~50 minerals with synthetic analogues. Their structure consists essentially of three-dimensional networks of Al/Si tetrahedra arranged in such a way so as to form channels containing water and exchangeable alkaline or alkaline-earth cations. Zeolite W is a synthetic MER-type analogue and its structure consists of double crankshaft chains (formed by alternating four-membered rings with four tetrahedral apices pointing upwards and downwards) which are filled with potassium ions and water molecules (Armbruster, 2001). Organophosphorous pesticides are very toxic agricultural chemicals and used for the protection of plants. Dimethoate, methylparathion, phosphamidon, fienitrothion, malathion, monocrotophos and phorate are most common organophosphorous pesticides and malathion is one of the most toxic and persistent organophosphorus pesticides which has caused serious environmental problems (Senthilkumaar, 2010 and Mohamed, 2009). For removing the pesticides, there are various methods such as advanced oxidation processes (Saritha, 2007), aerobic degradation (Rajashekara Murthy, 2007), photocatalytic degradation (Aungpradit, 2007), combined photo-Fenton and

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biological oxidation (Ballesteros Martin, 2008), nanofiltration membranes (Ahmad 2008), ozonation (Maldonado, 2006), and adsorption (Naushad, 2013). Malathion is usually used by Egyptian farmers, therefore its removal from water sources can become a very important problem. Malathion was studied on bagasse fly ash and found to be 0.006 µmol/g (Gupta, 2002).

Experimental

Materials and Instrumentation

Potassium hydroxide (Merck), Alumina (Merck), Colloidal Silica (Merck) and deionized water were used for synthesis of Zeolite W. An infrared spectrum of the synthesized zeolite was recorded by "Spectrumto-Perkin Elmer" spectrophotometer in the range of 4000-400 cm^{-1} by using KBr pellets. X-Ray diffraction spectra were recorded by using "Miniflex 600" Diffractometer. The operational detailed technique was set as follows: Copper Kα radiation at 40 kV30mA, with a goniometry speed of 2°/min and a step of 0.02° in the 2θ range scanning from 5° to 90° for synthesized powder samples. SEM micrographs were obtained using the ESEM quanta 200 FEI, SEM–EDAX instrument at 20 000× magnification.

Synthesis of Zeolite W

12.4 g of potassium hydroxide and 5 g alumina were dissolved in 20 ml of distilled water and stirred while heating a gentle boil until dissolved. The solution was then cooled at room temperature and distilled water was added to attain the original weight. The solution formed was labeled as 'A'. 18.5 ml of distilled water and 24.2g colloidal silica were mixed into the solution of 'A'. The final solution was poured into 125 Teflon Lined Autoclave and kept in an oven at 150^o C for 48 hrs for crystallization. After the stipulated contact time autoclave was taken from the oven and quenching of the autoclave was done. The formed zeolite W was washed and dried at 110^o C.

Adsorption Experiment

The adsorption of the methoxychlor onto zeolite W was investigated by batch experiment. Batch sorption studies were carried out at desired pH value, different concentration of sorbent dosages in 150ml stoppered conical flask containing 50 ml of test solution. 200 mg of adsorbent material was then added and the content of the flask was shaken for 24 h in an electrically thermo stated reciprocating water bath shaker at the rate of 110strokes/min at 30°C. After attaining the equilibrium, the content of the flask were filtered using Whatman filter paper 42 which separated the content of the flask into solid and solution phases. The filtrate content was centrifuged at 15000 r.p.m. at same tempretaure to restrict the interference of any suspended nanoparticles. The concentration of malathion in the filtrate was evaluated spectrophotometrically. The sorbed concentration of malathion on to synthetic zeolite at equilibrium was calculated by the difference in the initial amount of the malathion (C_i) and after shaking (C_e). The sorption percentage of malathion was calculated as follows:

$$\% = \frac{C_i - C_e}{C_i} \times 100$$

RESULTS AND DISCUSSION

Scanning Electron Microscope- Energy Dispersive XRay Spectroscopy (SEM-EDX): The SEM micrographs represent the crystalline morphology of zeolite W in the form of square pillar crystals and needle like crystals of 9.9 nm average length under 20000 may having particle size of 5µm. these results were in good confirmation with the reported data (Jin Hou, 2012). The elemental composition of zeolite W is 3 K_2O: 0.05 Na_2O : Al_2O_3 : 5 SiO_2 : 100 H_2O which has been confirmed by EDX technique.

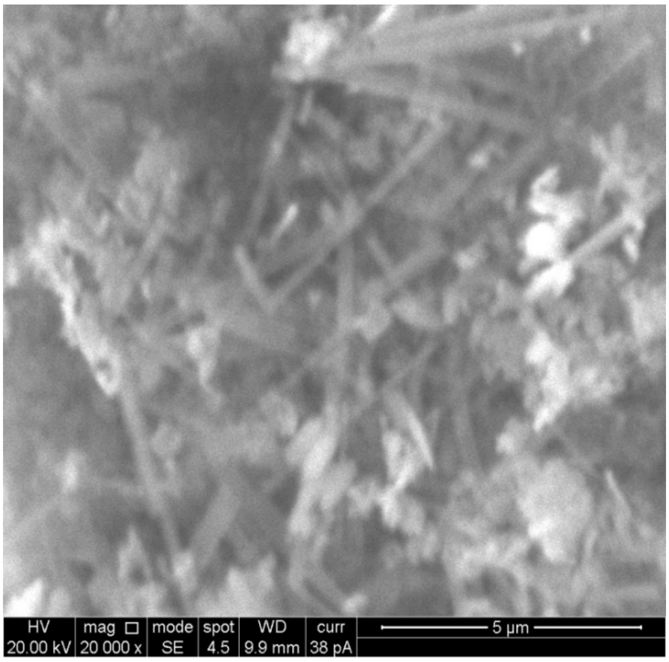

Fig. 1. SEM micrograph of Zeolite W

XRD

The X-Ray Diffraction pattern shows the crystalline phase of zeolite without any amorphous phase. The X-ray diffraction peaks of the synthesized have been found to be 12.5, 27.4 and 36.2, which are the characteristic peaks of zeolite W **[25]**. Fig (3) shows the X-ray diffraction pattern of synthetic template free zeolite powders. This confirm that the synthetic sample is zeolite W.

FTIR analysis

FT-IR spectra of Zeolite W are shown in Figure 4. The asymmetric stretching vibration of internal tetrahedra band of Si-O-Al at 1037.51 and the symmetric stretching vibration and bending vibration modes of Si-O-Al bonds (TO_4 tetrahedra) were observed at 637.93 and 591.38 cm^{-1} band. The absorption band at 1633.16 cm^{-1} is due to deformation vibration of OH groups. A valence vibration of OH group was observed at 3436.38 cm^{-1}.

Effect of adsorbent amount

Amount of adsorbent is essential parameter as it determines the adsorption capacity of an adsorbent for a given initial concentration (Naushad, 2014). The effect of adsorbent amount on the adsorption of malathion was studied at different doses (0.05–0.2 g) of zeolite W. It is clear from Fig 5 that the adsorption of malathion increases with the increase in adsorbent dose. After doses of 200 mg no change was seen in adsorption percentage of malathion which might occur because of the overlapping of active sites at higher doses. 0.2 g adsorbent dosage results in optimum adsorption.

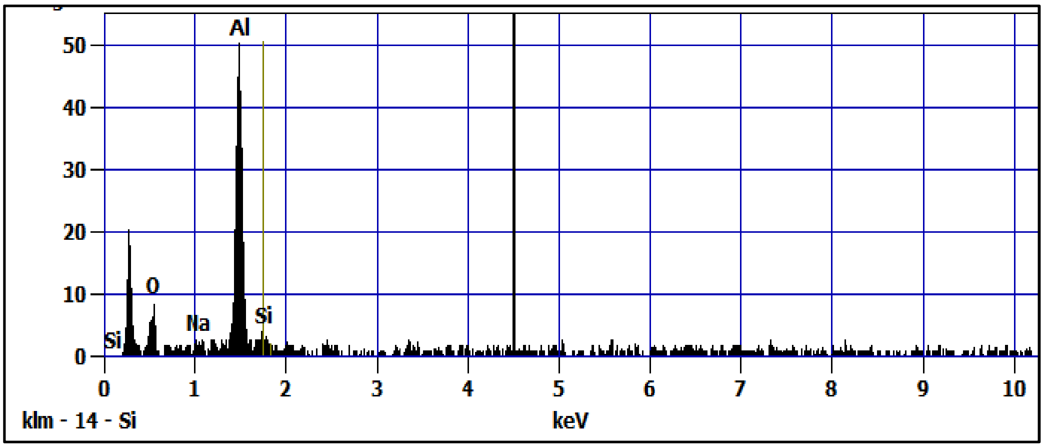

Fig. 2. EDX graphs of Zeolite W

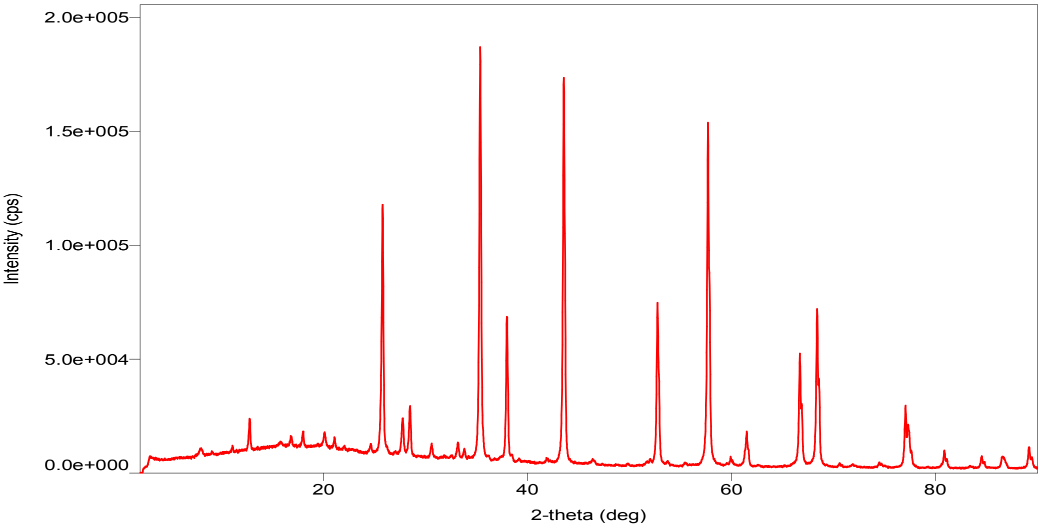

Fig. 3. XRD of Zeolite W

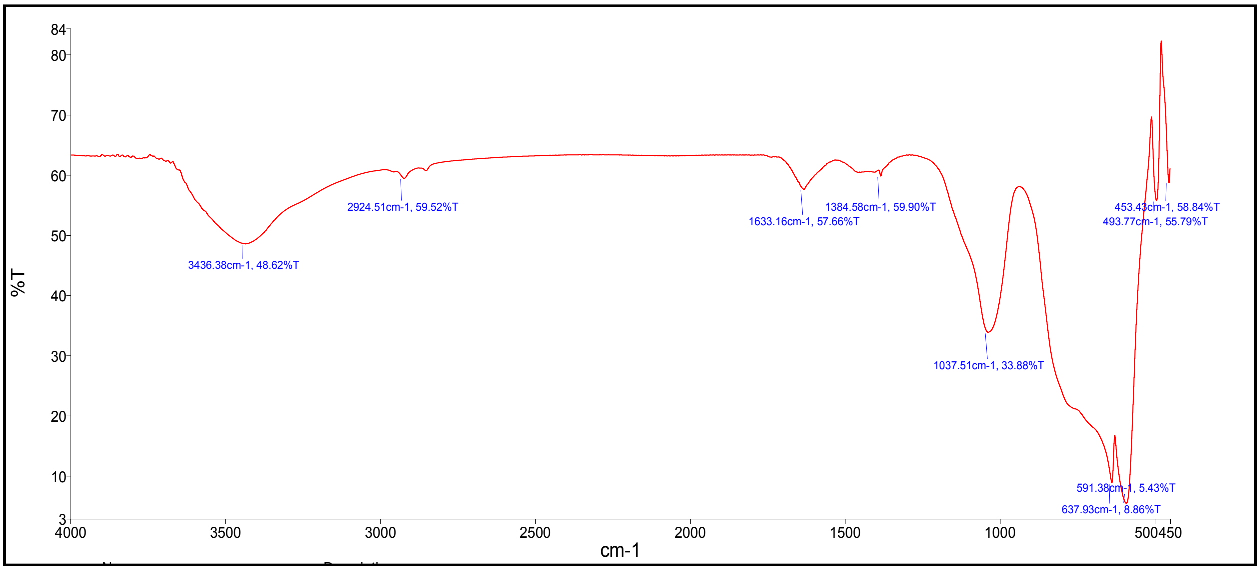

Fig. 4. FT-IR of Zeolie W

Effect of pH

The solution pH plays a significant role during adsorption process as it influences solute solubility and uptake capacity over the adsorbent surface (Issa, 2014). The effect of pH on the adsorption process depends on the type of adsorbent material, and its individual effect on the malathion. However, malathion decomposes in both acidic and alkaline environments, therefore, in this study, the pH was considered in the neutral range (Bojemueller, 2001). In this investigation, pH studies were carried out in pH range of 3–11 for 24 h. The pH of the solution was then adjusted using NaOH/HNO_3 solutions.

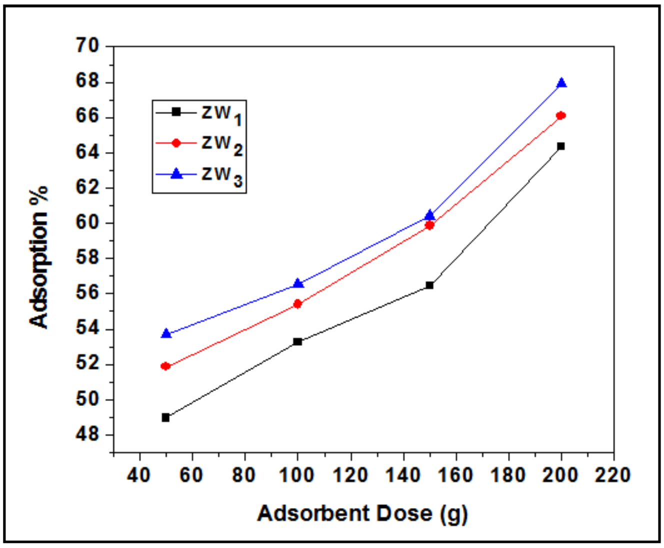

Fig. 5. Effect of adsorbent dose on sorption of malathion onto zeolite W

It is apparent from Fig.6 that the adsorption of malathion was increased with the increase in pH and highest percentage of adsorption of malathion was found at pH 7. Beyond pH 7, the sorption became constant.

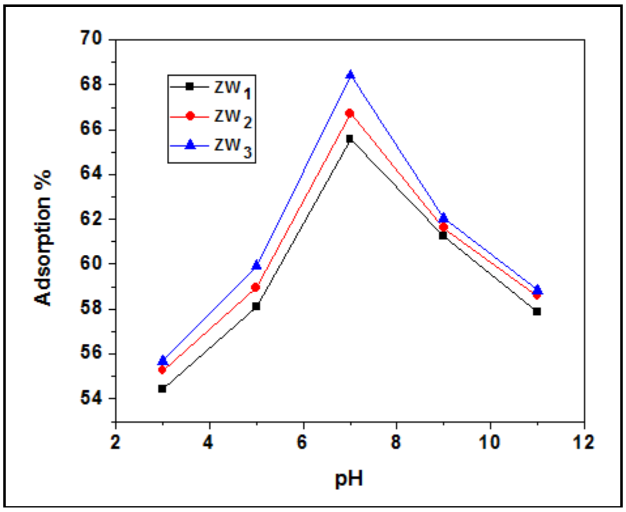

Fig. 5. Effect of pH on sorption of malathion onto zeolite W

Effect of Temperature

Effect of temperature on sorption of malathion on Zeolite W was shown in Fig 6. Different temperature effect has been studied against adsorption of malathion. An increase in temperature from 303.15 K to 323.15 K results in a decrease of sorption of pesticide by all the materials. The decrease in adsorption is due to the change in the energy of adsorption or weakening of vanderwaals forces of attraction between the pesticide and mineral surface causing in physical sorption of pesticide with a change in temperature (Deepesh Bhardwaj, 2007).

Effect of time

Contact time is a key factor in designing a treatment process with minimum time and maximum efficiency. Some research on organophosphorus pesticides removal showed an increased efficiency by increasing the contact time (Lertpaitoonpan, 2009). In Fig. 7, adsorption data show that the quantity of malathion adsorbed per gram of adsorbent raises when equilibration time increases. The graph of the quantity of malathion adsorbed per gram of adsorbent in comparison with the contact time between adsorbate and adsorbent shows that the adsorption gets asymptotic for the time axis after 12 hours of equilibration.

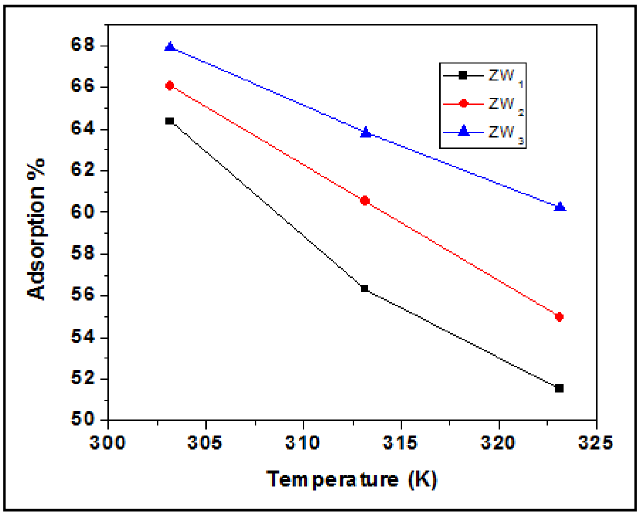

Fig. 6. Effect of temperature on sorption of malathion onto zeolite W

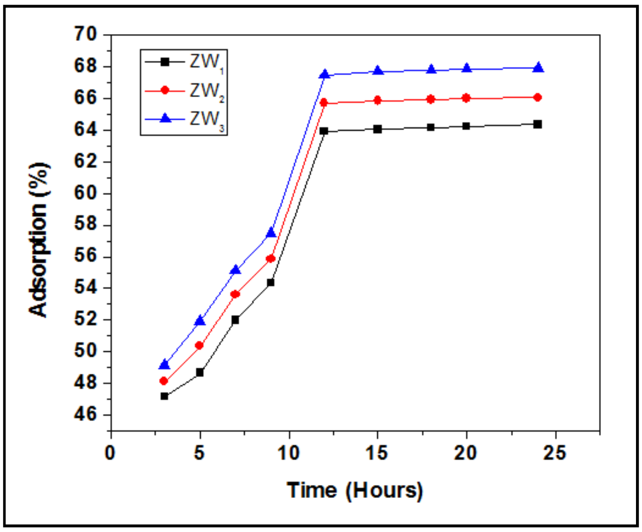

Fig. 7. Effect of time on sorption of malathion onto zeolite W

It represents approximately an equilibrium pattern. As the maximum adsorption is observed for all adsorbents at 24 of equilibration, the optimum equilibration time is 24 hours for other adsorption studies is considered.

Conclusion

The selected aluminosilicate namely Zeolite W is established to be probable adsorbents for the adsorption of malathion from aqueous solutions. Synthesized zeolite-W was characterized with different techniques. Adsorption of malathion over this synthesized material was carried out successfully. Adsorption of malathion was found to be high initially. It was observed that adsorption increases with increase in the contact time only up to 12 h later than it remain nearly constant. Further desorption experiments were also achieved successfully and desorption pattern discovered that desorption has happened from both weak as well as strong adsorption sites. The adsorption of malathion is pH dependent and its adsorption capacity increased with increasing the pH up to 7. Beyond pH 7, the sorption became constant. Zeolite W used to eliminate the malathion successfully from aqueous solutions in the concentration range of (0.05 – 0.2 g).

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