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KINETICS AND ISOTHERM STUDY FOR ADSORPTION OF METHYL ORANGE DYE INTO LOW COST AGRICULTURAL BY-PRODUCT (OLIVE SOLID WASTES) AS ADSORBENT

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ABSTRACT

The removal of dyes from wastewater is a matter of great interest in the field of waste water treatment. The effluents from many industries often contain one or more toxic dyes. methyl orange (MO) is one of the most common water-soluble dyes. Therefore, an increased interest has been focused on removing such dyes from wastewaters. In general, several methods are used for the removal of dyes from wastewater. Among the treatment methods, adsorption is comparatively superior. Agricultural wastes are renewable and available abundantly at no or low costs. Due to the abundant availability at no or low costs, little processing cost and ability to biodegrade Olive Solid Wastes (OSW) materials, an agricultural by-product, were identified as a potential adsorbent material for wastewater treatment in this study. Batch experiments were carried out for the sorption of methyl orange onto Olive Solid Wastes (OSW) particles. The operating variables studied were initial solution pH, initial dye concentration, and OSW dose. Equilibrium data were fitted to Freundlich isotherm equation. The monolayer sorption capacity of OSW for methylene blue sorption was found to be 101 mg/g. The sorption kinetics was found to follow pseudo second order kinetic model.

***Keywords**:*
Olive solid wastes.
Methyl orange.
Kinetic model.
Isotherm model.

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1 INTRODUCTION

Freshwater is already a limiting resource in many parts of the world it will become even more limiting due to increased population and climate change [1]. This limitation will be caused not just by increased demand for water but also by pollution in freshwater ecosystems pollution decreases the supply of used water and increases the cost of purifying [1].

Fresh water from rivers lakes and aquifers used to meet the global consumption has increased rapidly in the last few decades. Due to anthropogenic water consumption, the annual runoff in many river basins decreased by more than 5% during 1971 to 2000. According to water research group it is estimated that global industrial water requirements would increase from 800 billion m^3 to 1500 billion m^3 by 2030 and the industrial withdrawals which accounts for 16% of today's global demand is expected to increase by 22% in 2030 [2]. It has been estimated that by 2025 approximately 60% of the total world population would live in regions potentially experiencing moderate to extreme live in regions potentially experiencing moderate to extreme water resource unavailability [2].

Dyes have been used as colorants at different industries such as textile, food, paper, cosmetic and textiles color their products. The discharge of coloured wastewater from these industries into natural streams has caused many significant problems such as increasing the toxicity and chemical oxygen demand (COD) of the effluent, and also reducing light penetration, which has a derogatory effect on photosynthetic phenomena. Researches indicate that approximately 15% of produced synthetic dyes per year have been lost during processing operations that involve the production and handling with many organic compounds hazardous to human health[3]. Wastewaters of dye production and application industries present an environmental problem because of the aesthetic nature due to the fact that the color is visible even in a low dye concentration. The textile industry consumes large quantities of water at its different steps of dyeing and finishing, among other processes. The non-biodegradable nature of dyes and their stability toward light and oxidizing agents complicate the selection of a suitable method for their removal. In addition, toxicity bioassays have demonstrated that most of them are toxic [3].

Dyes are human made organic colorants used by various industries such as textile, painting, and cosmetics in manufacturing colored products most of these dye contain aromatic ring in their structure that could be harmful to human life considering mutagenic and carcinogenic properties. Several research reports revealed that very small quantities of dyes are highly toxic which causes acute disorders in aquatic organisms [4]. Cationic dyes impose serious health hazards to the environment and human being and are known to cause skin and eye irritation, increase heart rates, vomiting upon digestion, coronary vasoconstriction, decreased cardiac output renal reproductive system dysfunction as well as damage to central nervous system [4].

Several methods such as membrane, electrochemical coagulation/flocculation (Papic et al., 2004), biological, etc. have been used for dye removal from wastewater. Many physicochemical methods have been tested, but only that of adsorption is considered to be superior to other techniques. This is attributed to its low cost, easy availability, simplicity of design, high efficiency, and ease of operation, biodegradability, and ability to treat dyes in more concentrated forms [4]. Adsorption is the process through which a substance originally present in one phase is removal from that phase by accumulation at the interface between that phase and a separate phase. Adsorption can occur at any solid fluid interface gas-solid interface or liquid-solid interface substance thus adsorbed on the surface is called the adsorbats and the substance on which it is absorbed is known as adsorbent [4].

Activated carbon, also called activated charcoal or activated coal, is a form of carbon that has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions[5]. However, the cost of activated carbon is relatively high which limits their usage. Cost-effective alternative technologies or adsorbents for this purpose are needed. Natural materials that are available in large quantities or certain waste products from industrial or agricultural materials may have the potential as inexpensive adsorbents. Agricultural wastes are renewable and available abundantly at no or low cost [5]. The preparation of low-cost adsorbents from waste materials has several advantages, mainly of economic and environmental nature. A wide variety of low-cost adsorbents have been prepared from different waste materials utilizing agricultural as well industrial and municipal wastes [6, 7]. Generally, agricultural wastes are cheap and abundant adsorbate materials that have interesting properties from the standpoint of removing dye from its aqueous solutions. Interestingly, their low cost makes them a good choice for water treatment [6-8].

The aim of this present work was to find out the capacity of olive stone waste(OSW) material, as as low cost alternative adsorbent in the removal of methyl orange(MO) from its aqueous solution. The primary objective of this research was to explore the potential of olive stone waste as a low cost agricultural natural adsorbent waste for methyl orange dye removal from its aqueous solution. The effect of initial solution pH, initial dye concentration, contact time, on methyl orange adsorption was investigated. Moreover, this paper also discusses different kinetic and isotherm studies were conducted to evaluate the adsorption capacity of olive stone waste.

2 MATERIALS AND METHODS

Adsorbent

Libya is one of the Mediterranean see countries producing high amounts of olive oil. This production generates a significant amount of olive solid wastes (OSW), which consists of skin, pieces of pit, and pulp of the olives and a small amount of olive oil. The utilization of OSW as a low-cost sorbent is therefore beneficial for in removal of methyl orange from its aqueous solutions by adsorption process. The OSW was obtained from a traditional olive oil extraction plant in Sabratha, Libya. It was washed several times with distilled water to remove impurity such as sand then dried in an oven at 65^{o}C for 1day. Dried (OSW) were ground by using a crusher. The resultant powders were passed through British Standard Sieves and particles below 350µm collected in a plastic container and used as adsorbent for adsorption experiments.

Adsorbate and Other Chemicals

The basic cationic dye methyl orange (MO) is a commonly used an anionic mono-azo dye in laboratory assays textiles and other commercial products it is thought to be toxic if swallowed or inhaled and avoid skin contact with the solution or the solid. The characteristics of these dye compounds have generated some challenging environmental problems such as increasing the chemical oxygen demand of wastewater thus reducing the percentage of light penetrating into the water which reduces photosynthesis activity in aquatic media[8] and molecular formula is C14H14N3NAO3S and the (MO) molecular weight is 327.33 g / mol [8]. Methyl orange (MO) is supplied by Sigma-Aldrich Pty. Ltd, Germany.

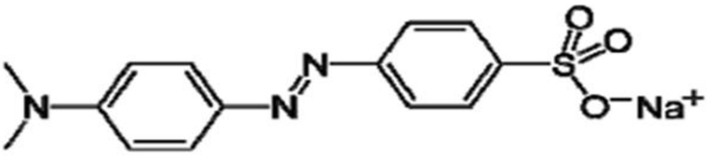

Figure 1.Chemical Structural of Methyl orang Dye [8]

A stock solution of 1,000 mg/l was prepared by dissolving the appropriate amount (1,000 mg) of MO in a litter of deionised water. The working solutions were prepared by diluting the stock solution with deionised water to give the appropriate concentration of the working solutions. The pH of the solutions was adjusted by addition of either 0.1 M HCl or 0.1 M NaOH solutions, respectively. All sample bottles and glassware were cleaned and then rinsed with deionised water.

The SP-8001 UV/VIS spectrophotometer was used to determine the concentrations of methylene blue dye in solution. pH measurements were done using Orien pH meter. The concentration of the residual dye was measured using UV/visible spectrometer at a λmax corresponding to the maximum adsorption for the dye solution by withdrawing samples at fixed time intervals, centrifuged and the supernatant was analysed for residual MO. Calibration curve was plotted between absorbance and concentration of the dye solution to obtain absorbance–concentration profile.

Adsorption Experiment

Adsorption measurement was determined by batch experiment of known amount of the adsorbent with 50 ml of aqueous methyl orange solutions of known concentration in a series of 250 ml conical flasks. The mixture was shaken at a constant temperature using at 150 rpm at 25°C temperature for 120 min. At predetermined time, the bottles were wither drawn from the shaker, and the residual dye concentration in the reaction mixture was analysed by centrifuging the reaction mixture and then measuring the absorbance of the supernatant at the wavelength that correspond to the maximum absorbance of the sample. Dye concentration in the reaction mixture was calculated from the calibration curve. Adsorption experiments were conducted by varying initial solution pH, adsorbent dose, initial methyl orange dye concentration and contact time under the aspect of adsorption isotherm and kinetic study.

3 THEORY AND CALCULATION

3.1 Dye Adsorption study

The amount of dye adsorbed OSW powder at time t is q_t (mg/g) which was calculated by the following mass balance equation [1]

$$qt = \frac{(Co - Ct)}{m} * V \qquad (1)$$

And the dye removal efficiency, i.e. % of Adsorption was calculated as:

$$\% \, Removal = \frac{(Co - Ct)}{Co} * 100 \qquad (2)$$

where C_o is the initial dye concentration (mg L^{-1})), C_t is the concentration of dye at any time t, V is the volume of solution (litres) and m is the mass of OSW powder in grams.

3.2 Dye Adsorption Kinetic-study

The controlling mechanisms of adsorption process such as chemical reaction, diffusion control or mass transfer coefficient are used to determine by kinetic models. The kinetics of dyes adsorption onto adsorbent materials is prerequisite for choosing the best operating conditions for the full-scale batch process. Thus, the kinetics of anionic and cationic dye onto various adsorbent materials was analyzed using different kinetic models which are presented below [1].

3.2.1 Application of Lagergren pseudo-first-order model on dye adsorption.

The linearized integral form of the Pseudo-first-order Model generally expressed as:

$$\log(q_e - q_t\ ) = \log\ q_e \frac{K_1}{2.303} t \qquad (3)$$

Where; q_t and q_e are the adsorption capacity at time t and at equilibrium, respectively (mg g^{-1}), k_1 is the rate constant of pseudo-first-order adsorption (min^{-1}) and t is the contact time (min). To plot log (q_e – q_t) versus t give a linear relationship from which k_1 and predicted q_e can be determined from the slope and intercept of the plot, respectively [1].

3.2.1 Application of Lagergren Pseudo-second-order Model on dye adsorption.

The adsorption kinetic may be described by the pseudo-second-order model [21]. The simplified and linearized to get described as :

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \qquad (4)$$

Where; k_2 (g (mg min^{-1})) is the second-order rate constant of adsorption.

The second-order rate constants were used to calculate the initial sorption rate, given by the following equation:

$$h = k_2 q_e^2 \qquad (5)$$

The plot of t/q_t versus t shows a linear relationship. Values of k_2 and equilibrium adsorption capacity q_e were calculated from the intercept and slope of the plot of t/q_t versus t according to Eq. (4).

3.3 Application of Adsorption Isotherm Models on dye adsorption.

The adsorption isotherm is significant for the explanation of how the adsorbent will interact with the adsorbate and give an idea of adsorption capacity. They play an important role to understanding the mechanism of adsorption. The surface phase may be considered as a monolayer or multilayer. Several isotherm models are presented in the literature. Langmuir and Freundlich models are the most widely used to describe the adsorption isotherm [1].

3.3.1 Langmuir Adsorption Isotherm Model to dye adsorption.

The Langmuir adsorption isotherm model assumed that adsorption takes place at specific homogeneous sites within the adsorbent, and it has been used successfully for many adsorption processes of monolayer adsorption. The linearized form of Langmuir isotherm that can be written as [1]:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_a q_m} \qquad (6)$$

Where,q_e is the amount of dye adsorbed at equilibrium time (mg/g),C_e is equilibrium concentration of dye in solution (mg L^{-1})), q_m is maximum adsorption capacity (mg/g) and Ka is isotherm constants for Langmuir (L mg^{-1}).

The slop and intercept of plot between Ce /qe vs Ce will giveq_m and Ka respectively.

3.3.2 Freundlich Adsorption Isotherm Model to dye adsorption.

The Freundlich adsorption isotherm model considers a heterogeneous adsorption surface that has unequal available sites with different energies of adsorption. The linearized form of Freundlich can be expressed as [1]:

$$Lnq_e = LnK_f + \frac{1}{n}(LnC_e) \qquad (7)$$

Where q_e is the amount of metal ion adsorbed at equilibrium time (mg/g), Ce is equilibrium concentration of dye in solution (mg L^{-1}). K_f is the capacity of the adsorbent and n is the intensity of adsorption constant for Freundlich. The plot of *Ln*q_e versus *Ln*Ce is employed to determine the K_f and n from intercept and slope respectively. Generally, the value of the linear regression correlation coefficient R^2 gives the indication which the model can be chosen to give best-fit.

4 RESULTS AND DISCUSSION

This section may be divided into two subsections.

4.1 Concentration calibration curve.

The cationic dye, Methyl orange dye of an analytical grade is used as the model adsorbate in this research study. Methyl orange has dark orange color and when diluted with water, the solution turns into light orange due to presence of oxygen. A stock solution of 1000 mg/L was prepared by dissolving 1g of MO powder in 1000 ml volumetric flask and filled it with ultra-pure water. Various dye concentrations 10,15,20,25 mg/L were prepared by diluting the stock solution with ultra-purewater. The spectrophotometer SP-2800 UV/VIS was used to measure the concentration of MO dye in solution where the maximum adsorption for the dye solution was measured. The calibration curve was plotted between the absorbance and concentration of MO dye solution to obtain the linear calibration equation as shown in Fig 2. The concentration of the MO dye at any time will be measured from the calibration curve.

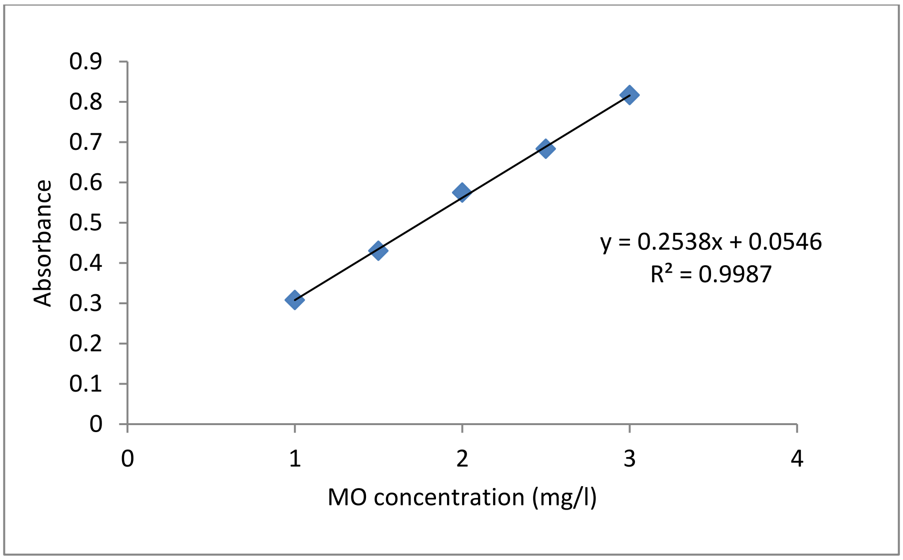

Figure 2. Calibration curve for absorbance at different MO concentrations and its linear regression.

4.2 Factors Affecting Adsorption of Dye.

4.2.1 Effect of Initial Solution pH on MO Dye Adsorption

The initial pH of the MO dye solution was important t parameter which controlled the adsorption process, particularly the adsorption capacity. The efficiency of adsorption is dependent on the solution pH because variation in pH leads to the variation in the degree of ionization of the adsorptive molecule and the surface properties of adsorbent. Figure (3 and 4), show that the effect of initial pH (3 to 10) on amount of dye adsorption, qt (milligrams per gram). The amount of dye adsorption increases with as the pH decreases, it is expected that the cationic dye adsorption also increases due to decreasing negative surface charge of the adsorbent. The negative charge on OSW biomass increased with decreasing pH, which was reported by [6]. In addition, lower adsorption of MO at acidic pH might be due to the presence of excess H^+ ions competing with dye cations for the available adsorption sites [7].

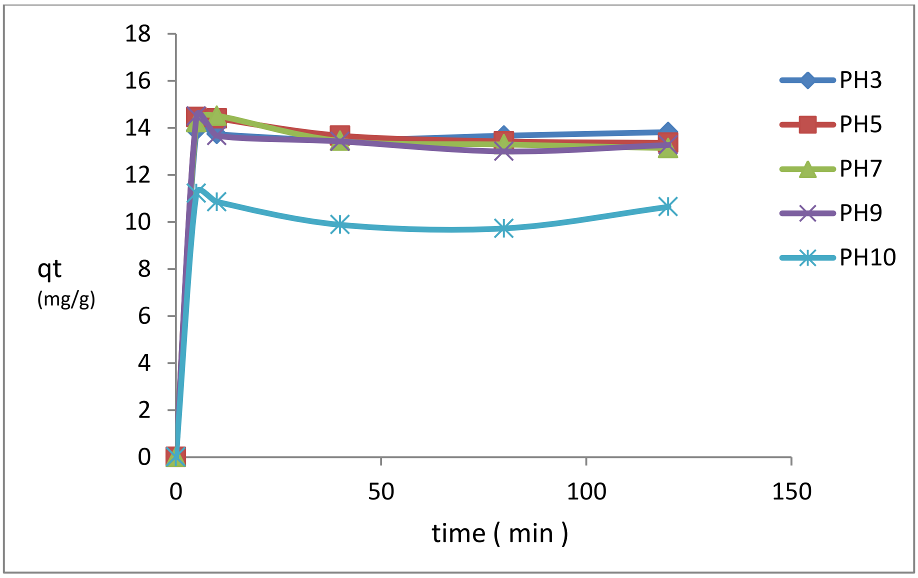

Figure 3. Effect of initial solution pH on the amount of adsorption of MO onto OSW.

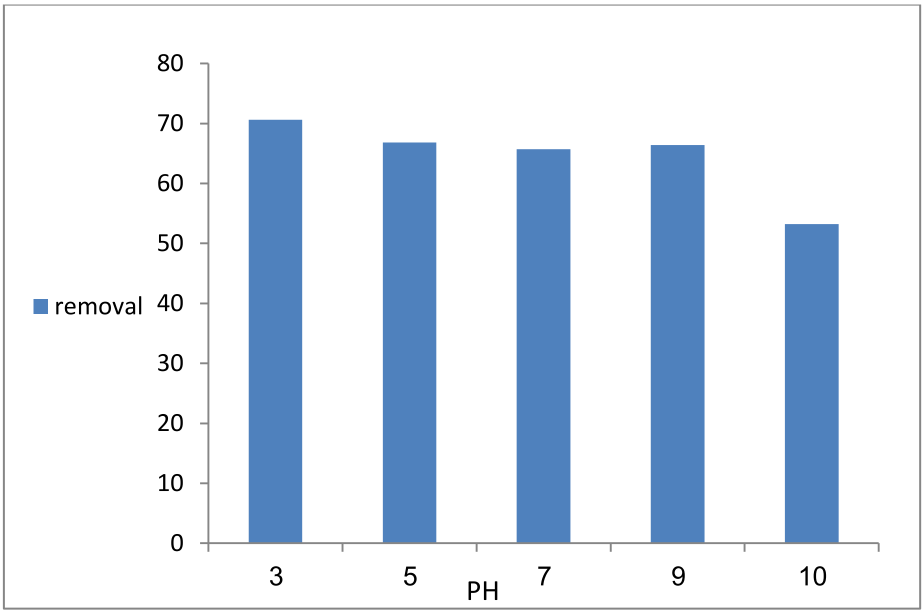

Figure 4. Effect of initial solution pH on percentage removal of MO onto olive waste.

4.2.2 Effect of Initial MO Dye Concentration on Adsorption

Figure (5 and 6) show that the increase at initial dye concentration leads to an increase in the adsorption capacity. As the initial dye concentration increases from 10 to 90 mg/L, the adsorption capacity of dye onto OSW from 14.12 to 138.3 mg/g. This indicates that the initial dye concentration plays an important role in the adsorption capacity of dye. However the % removal of dye decreased from 70.6 to 76.8 % on increasing the initial dye concentration from 10 to 90 mg/L after 120 minutes. Further, it was observed that the amount of methyl orange dye uptake, q_t mg/g, is increased with increase in initial dye concentration. Basically, from the figure, the adsorption percentage decreases and the extent of adsorption increases with increasing initial dye concentration. This is so because the initial dye concentration provides the driving force to overcome the resistance to the mass transfer of dye between the aqueous and the solid phase. For constant dosage of adsorbent, at higher initial dye concentration, the available adsorption sites of adsorbent become fewer, and hence, the removal of methyl orange depends upon the initial concentration [8]. The increase in initial concentration also enhances the interaction between adsorbent and dye. Therefore, an increase in initial dye concentration leads to increase in the adsorption uptake of dye. Kinetic experiments indicated that adsorption of MO on OSW followed three step processes, a rapid initial adsorption followed by a period of slower adsorption of MO and finally no high adsorption. Similar types of results are reported by various researchers for dye adsorption on melon husk Giwa *et al*. [8], on organoclays Hasseb *et al*. [9], and on waste activated carbon Sneha *et al* [10].

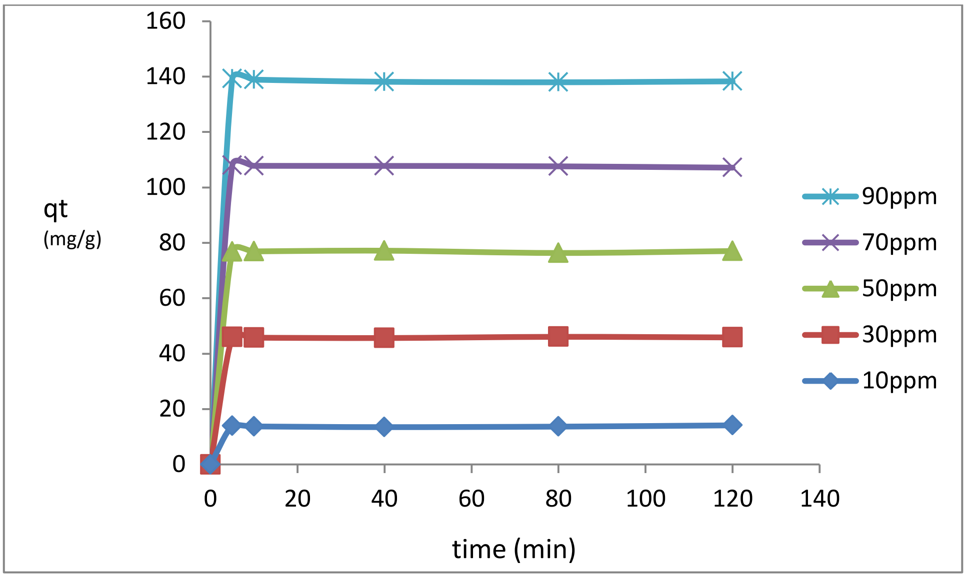

Figure5 Effect of initial solution concentration on percentage removal of MO onto OSW.

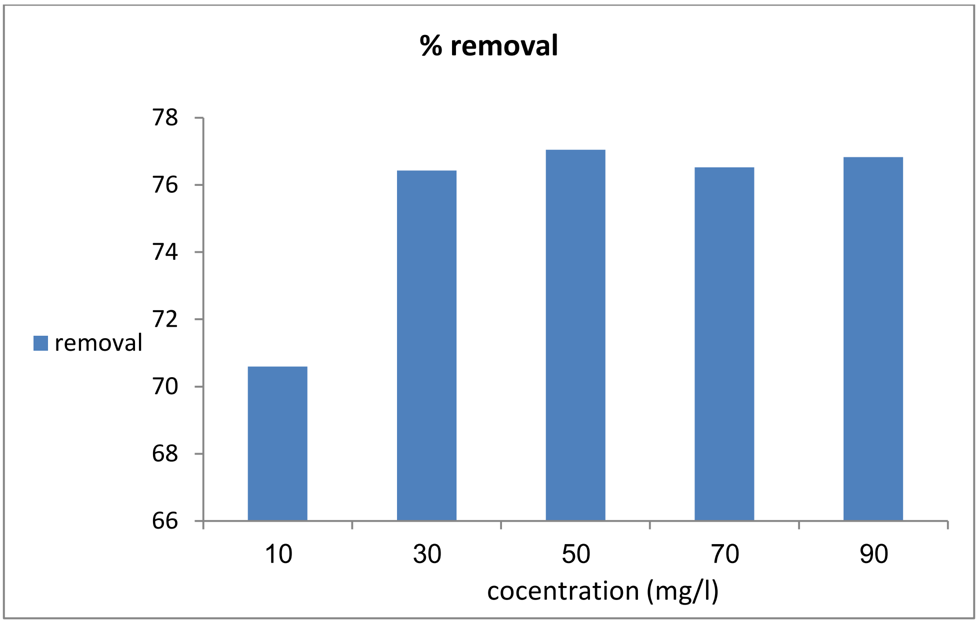

Figure 6. Effect of initial concentration on percentage removal of MO onto OSW

4.2.3 Effect of Adsorbent Dose on MO Dye Kinetic Adsorption

The amount of various OSW doses of 20 ,25 and 30 mg in the removal of MO dye represents an important parameter due to its strong effect on the capacity of an adsorbent at given initial dye concentration. Figure (7 and 8) show that at equilibrium, the increase in adsorbent dosage from 20 to 30 mg resulted in decrease of amount of adsorbed dye for both the systems. A fixed mass of OSW materials can only adsorb a fixed amount of dye. At higher OSW to methyl orange concentration ratios, there is a very fast superficial sorption onto the biomass surface that gives a lower dye concentration in the solution compared to the lower biomass to dye concentration ratio. This is because a fixed mass of biomass can only adsorb a fixed amount of dye. Therefore, the more the adsorbent dosages, the bigger the volume of effluent that a fixed mass of biomass can purify. In general, pH of final solution increases gradually with the increase in adsorbent dosages. It may be attributed to evident increase of the amount of negatively charged sites which can induce more H^+ ions adsorb on OSW surface and result in an increase in pH of the final solution. A similar behaviour was observed for methylene blue adsorption on on waste activated carbon Sneha *et al* [10].

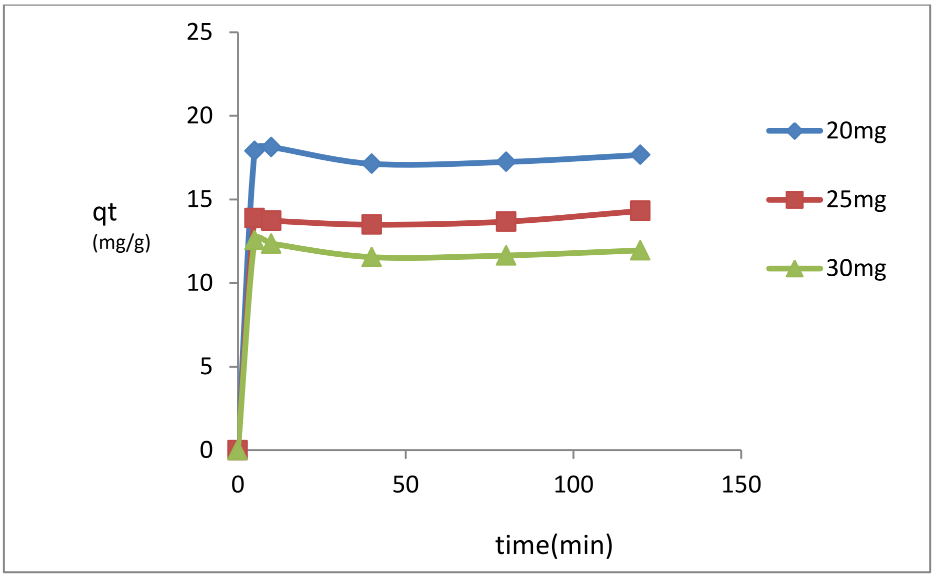

Figure 7. Effect of adsorbent dose on the adsorption of MO onto OSW

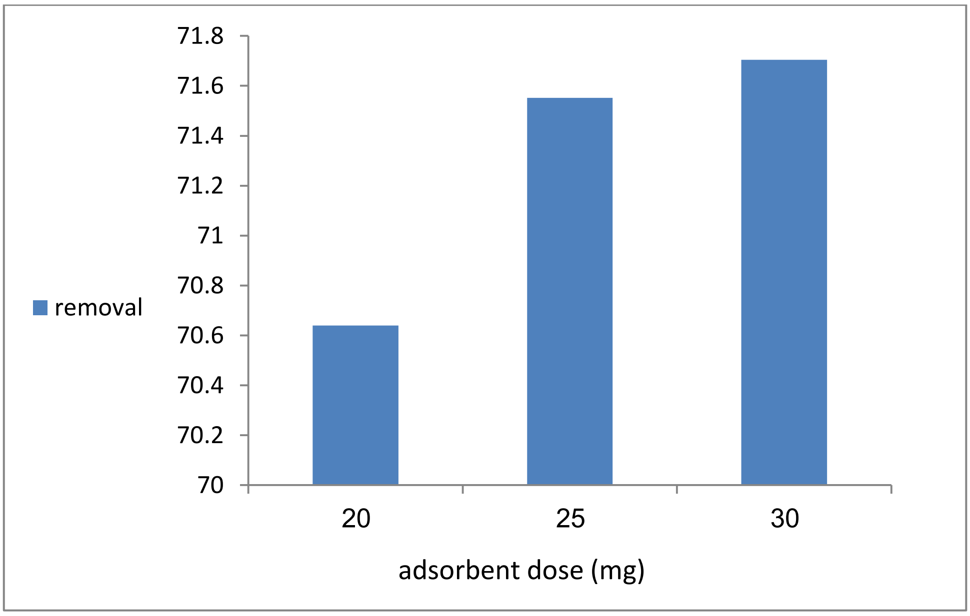

Figure 8. Percentage removal under Effect of adsorbent dose on the adsorption of MO dye on OSW.

4.3 Applications of Adsorption Kinetic Models

In order to understand the mechanism of adsorption rate of control and the transient behavior of MO dye, adsorption kinetics must be studied under physio-chemical process parameters such as initial MO dye concentration. Lagergren Pseudo-first-order and Pseudo-second-order kinetic models were used to determine the most suitable and practical model in term of high adsorption capacity and fast adsorption rate. Higher linear regression coefficient R^2 from pseudo-second-order kinetics model indicates the suitability of pseudo-second-order kinetics model over other model. In addition, the values of calculated qe from pseudo-second-order model are very close to the experimental qe value which gives more suitability of this model. While pseudo-first-order kinetic model gives a much lower value of the equilibrium qe than the experimental qe value, therefore this model is not applicable for this system Figure (9 and 10). Similar kinetic model parameters were obtained by different researchers for a few other systems reported in the literature Indrajit *et al* [11] and Gietu *et al* [12].

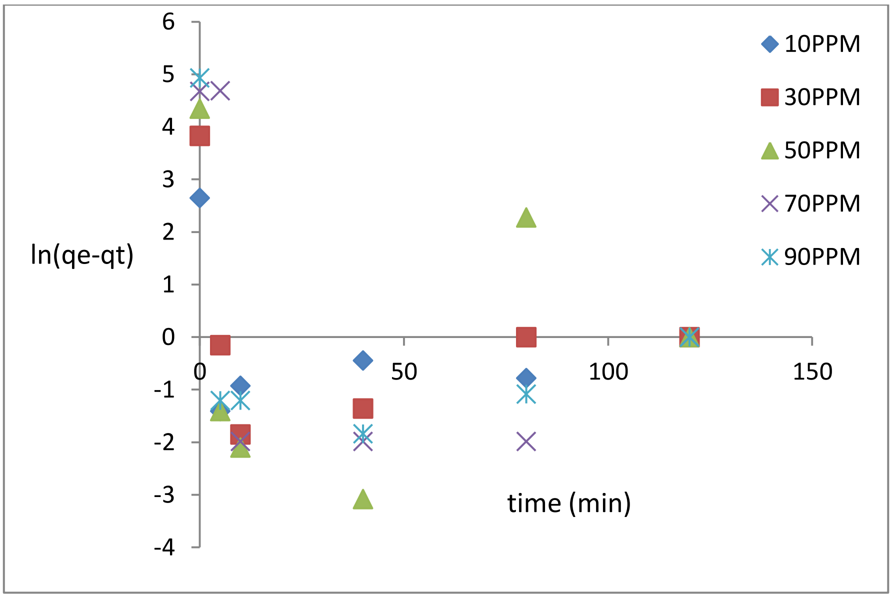

Figure 9. Pseudo-first order plot for adsorption of MO onto OSW at different initial MO concentration

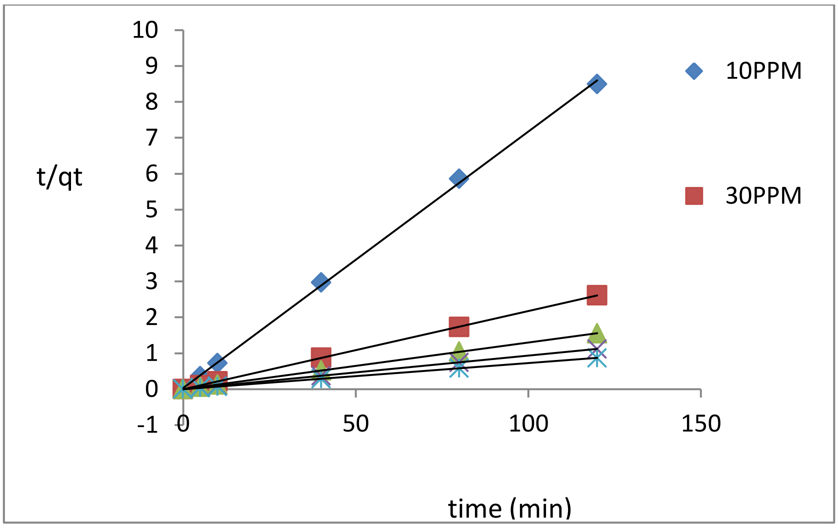

Figure10. Pseudo-second order plot for adsorption of MO onto OSW at different initial MO concentration

4.4 Adsorption Equilibrium Isotherm Models

Equilibrium isotherm studies are important to describe how MO dye molecules interact with the OSW surface and determine the max adsorption capacity of adsorbent. The applicability of the isotherm equation is compared by judging the correlation coefficients, R^2. The theory of Langmuir and Freundlich models is presented in section (3.3). Figure (11) showed Langmuir isotherm fittings for OSW adsorbent with low linear regression. The maximum monolayer adsorption capacity of OSW, q_m, and constant related to the binding energy of the sorption system, Ka, is calculated from the slop and intercept of this plot. The maximum monolayer adsorption capacity q_m of OSW was 101 mg/g and the constant related to the binding energy of the sorption system Ka was 1.0 L/g.

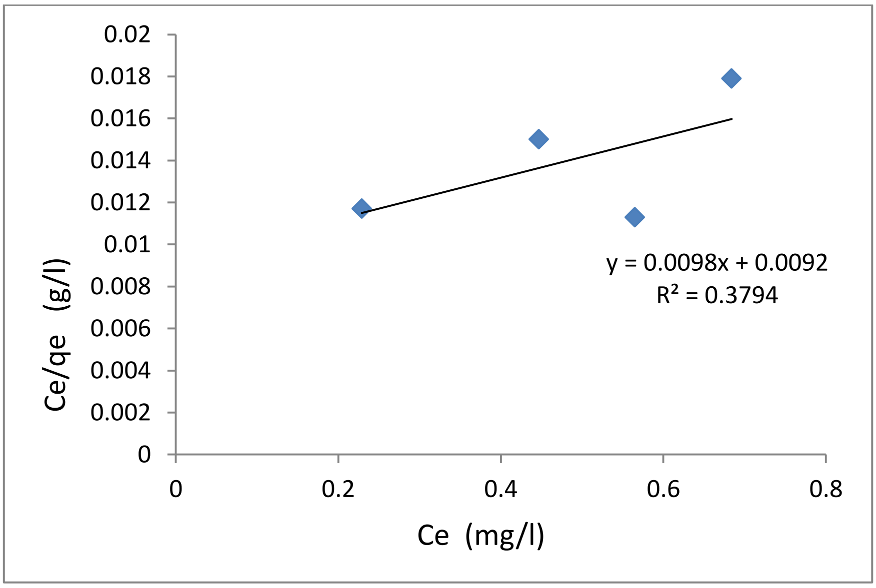

Figure 11. Langmuir plot: amount of adsorbent (OSW)

On the other hand, Freundlich isotherm fittings for OSW adsorbent are shown in Figure (12). The linear correlation coefficients R^2. Freundlich constants, rate of adsorption, n and adsorption capacity K_f, are calculated from these plots, which is 1.656 and 56.656 mg/g, respectively for OSW.

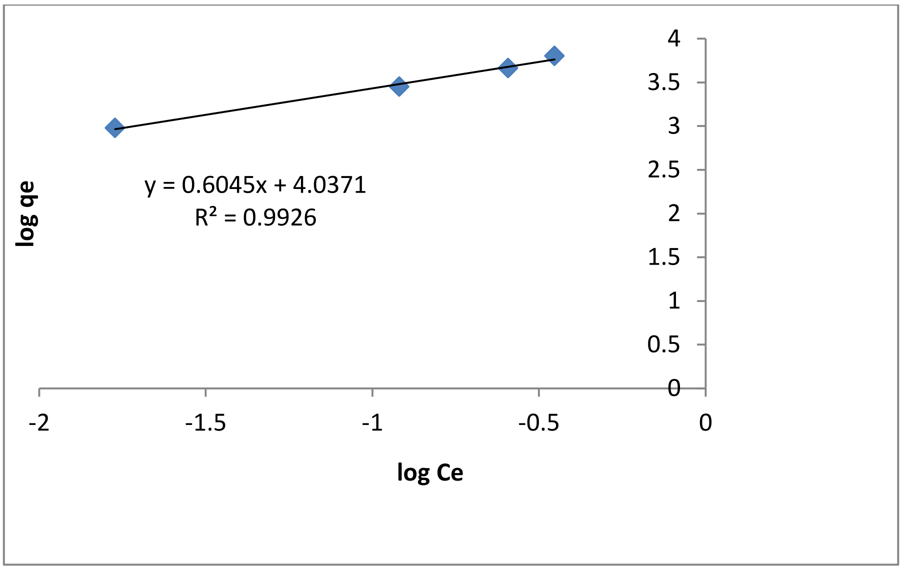

Figure 12. Freundlich plot: amount of adsorbent (OSW)

R^2 values indicate the suitability of Freundlich equation isotherm model for their experiment data. It was also found that the adsorption equilibrium data fit for Freundlich equation with a correlation coefficient value of (0.9926). This means that the reaction was heterogeneous behaviour. Several studies have been investigated on the removal of dye from its aqueous solution using low cast adsorbents Anwar *et al* [6], Munagapati *et al* [4] and Nhung *et al* [3].

5 CONCLUSIONS

From this study, the ability of Olive Solid Wastes (OSW) to remove Methyl orange dye from its aqueous solution was investigated. We can conclude that the amount of MO removed by (OSW) increases with decreasing (OSW) dose, and pH solution, but the amount of MO removed by (OSW) increases with increasing initial MO dye concentration. The equilibrium adsorption isotherms have been validated in detail by Langmuir and Freundlich models. Equilibrium data fitted very well wite the Freundlich isotherm equation, confirming the monolayer adsorption capacity of methyl orange dye onto (OSW) with a monolayer adsorption capacity of 101 mg/g. The results also showed that the process follows by pseudo second-order kinetics model.

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دراسة الخواص الحركية والأزوثيرم لامتزاز صبغة الميثايل البرتقالي من خلال منتج زراعي ثانوي (*مخلفات* عصر الزيتون الصلبة) كممتز

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الملخص

تعتبر ازالة الصباغ من مياه الصرف الصحي مسألة ذات أهمية كبيرة في مجال معالجة مياه الصرف الصحي. غالبا ما تحتوي النفايات السائلة من العديد من الصناعات علي عدة اصباغ سامة. ميثيل البرتقالي هو احد الاصباغ القابلة للذوبان في الماء. لذلك، تم التركيز مشكل متزايد على ازالة هذه الاصباغ من مياه الصرف الصحي. بشكل عام، يتم استخدام عدة طرق لازالة الاصباغ من مياه الصرف الصحي. من بين طرق المعالجة، تعتبر طريقة الامتزاز متفوقة نسبيا. تعتبر النفايات الزراعية متجددة و متوفرة بكثرة بدون تكلفة او بتكلفة منخفضة. فقد تم اختيار مخلفات عصر الزيتون الصلبة، وهي منتج ثانوي كمادة ممتزة لمعالجة مياه الصرف الصحي. في هذه الدراسة، تم اجراء تجارب معمليه دفعيه لامتزاز صبغة الميثيل البرتقالي بواسطة مخلفات عصر الزيتون الصلبة، تحت عوامل تشغيلية مثل الاس الهيدروجيني للمحلول و كمية المادة الممتزة و كذلك تركيز الصبغة في المحلول. وتمت في هذه الدراسة مقارنة النتائج المعملية مع نماذج رياضية لحركية التفاعل و قد وجد ان العملية تتبع النموذج الحركي من الدرجة الثانية ويتبع معادلة(فرندلش) للاتزان و تبين ان العملية تكون بتفاعل غير متجانس.

الكلمات الدالة:
المثيل البرتقالي.
مخلفات عصر الزيتون الصلبة.
النموذج الحركي.
نموذج الاتزان.

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