Kinetic Isotherm of the Adsorption of Potassium Chloride on the Surfaces of Biochar Particles Prepared from the Solid Residues of the Olive Mill

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Abstract :
 The aim of this work, was to study the kinetics of adsorption of biochar particles prepared from the biomass of solid residues of the olive mill, in KCl solution, (KCl/BioC), system to be adopted to predict the default values of adsorption.

 The experiment included the use of the biochar as a solid phase and source for adsorption surfaces in this system, where 3 equations were tested to model the resulted equilibrium data, in order to test their suitability for application in this system .

 It was found that this adsorption kinetics isotherm follows a straight line positive relationship expressed very efficiently by first order equation, $(R2 = 0.996)$, then Freundlich logarithmic equation $(R2 = 0.968)$, within the limits of the experimental points of KCl concentrations in the liquid phase less than 60 meq/liter. But an application of a default second-order equation for this system proved to be more expressive for all values of concentrations, where the correlation coefficient for this equation recorded high accuracy $(R2 = 0.999)$, also it allows to predict any hypothetical value of adsorption for any KCl concentration in the solution including the maximum adsorption level.

Keywords: *Biochar, Adsorption, desorption, Olive mills, Freundlich, KCl/BioC system.*

I. INTRODUCTION

It is useful and necessary to describe the mechanism of adsorption/desorption of ions and salts on solid surfaces both quantitatively and mathematically to facilitate the interpretation of applications of this characteristic in agriculture such as adsorption of cations and anions in the soil and to prevent them from being washed and lost to groundwater, salts adsorption and alleviating their negative effects on plant roots, adsorption of toxins, gases and pesticides residues formed in the soil during the growing season, as well as purification of drinking water, sewage and agricultural water, and other applications. Also, it can be applied to describe the kinetics of the adsorption of salts into the solution on the solid surfaces of the biochar particles.

Biochar is defined as a granular material, rich in carbon, with high porosity, produced by what is called "pyrolysis" for organic materials (such as wood, manure, and crop residues) at temperatures (400-700 Co), under anaerobic conditions or little oxygen [1].

This product is used in many agricultural and environmental applications such as soil improvement, reserving a wide range of organic and inorganic materials such as polycyclic aromatic hydrocarbons ([2], [3]) and heavy metals ([4], [5]).

If the organic matter provides good growth conditions and nutrients for plants and microorganisms in the soil, the "biochar" acts as a reagent to absorb, hold and retain water and nutrients ([6], [7]).

Therefore, the application of biochar improves soil fertility by changing many of its physical and chemical properties such as the ability to retain water and increase porosity, aeration, specific surface area, soil pH, cation and Anion exchange capacity ([8], [9]).

Re. [10] studied the effect of biochar in reducing nitrate and potassium leaching from soil planted with sorghum, mentioned that, the lowest percentage of nitrate and potassium leaching was when applying 45 tons/h of biochar and achieving the highest growth rate of yellow maize plants as they found in the same year when using biochar in the soil of Anthrosol and Ferralsol after they were cultivated with beans, that biochar increased the proportions of elements N, P, Ca, Mn, Zn in the cowpea plants in both soil types, and nitrate leaching was very minimal in Anthrosol soil. Also, biochar application increased plant growth and availability of nutrients.

Re. [11] studied the role of biochar in improving the growth and yield of cultivated potatoes under salt stress conditions, concluded that biochar reduced sodium uptake by binding it on its adsorption sites,

and reducing osmotic pressure through enhancing soil moisture holding and nutrient release especially the ions (K +, Ca ++, Mg ++) in the soil solution.

On the other hand, some studies have demonstrated the efficacy of biochar in the adsorption and removal of heavy metals from water [12], and its movement in soil [13]. Where biochar has negative functional groups, distributed over the surfaces of its particles, and these groups increase over time, as a result of soil oxidation processes [14], as the formation of these functional groups, and the sites of adsorption on its surfaces, and inside the pores, increase the cation exchange capacity (CEC) [15], thus increasing the ability of soil treated with biochar to form complexes with heavy metal ions, and since the basic construction of biochar molecules is more stable in the soil environment [16], heavy mineral complexes, with biochar, is more stable than that formed with the organic matter in the soil.

Re. [17] found that, when the application rate of biochar reached 0.4%, the treated soil showed an improvement in the available water capacity (AWC), in sandy soils, while it had no noticeable effect on clay soils. Also re. [18] found that the treated clay soil with biochar prepared from wheat residues, showed more significant ability to prevent insecticides from being washed, compared to loamy soils treated with biochar, and found that biochar differs in its effect according to its source and the conditions of pyrolysis in one hand, and the soil type on another hand.

As solid waste disposal is a concern around the world, biochar production by pyrolysis is one of the modern and sustainable environmental methods for disposal of these wastes through recycling, and then used as anamendment to improve soil properties [19], due to the unique and specific characteristics of biochar [20].

It is noteworthy that the biochar specific characteristics , such as the area of its specific surfaces and its porous structure, depend mainly on the source of the raw materials and the method of preparation, such as temperature and duration of pyrolysis [9], where several studies have shown differentiations in biochar characteristics according to the source of the raw materials used in its preparation as found by [21], they indicated that the cation exchange capacity varies greatly between the types of the biochar prepared from different sources such as sawdust of eucalyptus and pine, sugar cane remains, coffee husks and poultry litter.

Hence, the aim of this work intended to study the ions adsorption/desorption kinetics of biochar prepared from the biomass of the solid waste of olive mill, as it is a widely available raw material in Syria, in order to assess its ability to improve soil properties.

II. MATERIALS AND METHODS

Locally prepared biochar from the biomass of the solid waste of olive mill, was used in this study, in order to test its adsorption capacity based on the specific surface of its particles.

A. Experimental preparations :

-1   The biochar was prepared locally in a traditional way by burning the biomass of the solid waste of olive mill, under anaerobic conditions, where the temperature rises to about 400 Co for an hour during which the biomass passes in several stages of drying, disintegration, thermal decomposition, and losing the volatile compounds, and gases combustion, then, most of the organic carbon is transformed into elemental carbon, (graphite), with some mineral impurities in the form of ash and calcium carbonate .

2-   After crushing the prepared biochar into fine particles (<1 mm), It was washed, dried and analyzed to identify some of its specifications according to table (1).

Table (1): Some selected specifications of the used biochar.

purity	Moisture	Or.C	AEC	CEC	pH$_{w1/10}$	EC$_{w1/10}$
%	%	%	Meq/100g	Meq/100g	-	mS/cm
86.2	2.6	4.24	33	217	9.3	0.33

3-Preparation the saturation solutions: A mother solution (0.1 N) of potassium chloride salt, (KCl), was prepared, and then the following concentrations of saturation solutions were prepared in a gradual order: 0, 4, 8, 16, 20, 25, 30, 35, 40, 50, 60, 100 meq/liter.

B. Experimental design and implementation :

-    The experiment was carried out in the laboratory of soil fertility and plant nutrition in Tishreen University. It included the saturation of biochar samples with different concentrations of potassium chloride solutions (KCl), according to table (2) in 3 replicates.

-    0.5) g) pre-washed biochar samples were taken into a (100 ml) polypropylene bottles .

-    Each sample was filtered and the filtrate was taken to measure the EC and calculate the concentrations of salts therein.

-    Concentrations of soluble salts represented by potassium chloride salt in the liquid phase of the biochar suspensions of type (0.5/100), were calculated for all treatments, by measuring the electrical conductivity (EC) in the filtrates according to [22], as shown in table (2).

-    Then, KCl saturation solutions were added to each bottle according to the treatments.

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The polypropylene bottles were placed on a mechanical shaker in horizontal rotation, at a speed of (150 r/m) at the laboratory temperature for a period of (24h), left for a week, and then put on the shaker again for another extra period of (1 hour).

III. RESULTS AND DISCUSSION

The study of the mechanism of kinetics of solutes between the liquid and the solid phases is an important work for modeling the adsorption systems and their kinetics, to get the right information about the homogeneity or heterogeneity of the adsorption process on surfaces and determining its applicability. In this regard, many models are used to describe the balanced data at equilibrium, assuming that adsorption takes place in a monolayer with equal and uniform forces on all sites, to be appropriate for Langmuir equation, which was found to be unsuitable for this system. Therefore, the Friendlich equation was applied in the expression and modeling of most kinetic data and adsorption relationships where it assumes unlimited adsorption sites and heterogeneous surfaces [23].

Therefore, Friendlich equation, was taken into consideration in this study totest its suitability for application in the case of studying the kinetic mechanism of adsorbed/desorbed salts between the liquid and solid phases at equilibrium in the KCl/BioC system, in comparison with other two models, one of which is First order equation, and the other is second order equation.

The values of the measurements of the salt concentrations in the filtrates before and after mixing and shaking process with biochar were calculated with their logarithmic values as shown in table (2).

Table (2): changes of the concentrations of adsorbed KCl on the specific surfaces of 100 g biochar particles under different KCl concentrations in the equilibrium solution.

No	Added KCl meq/l (C)	Log C	Adsorbed KCl meq/100g (X)	Log X
1	0	0.000	-8.81	-0.950
2	4	0.602	3.95	0.597
3	8	0.903	18.21	1.260
4	12	1.079	31.61	1.500
5	16	1.204	49.48	1.694
6	20	1.301	58.59	1.768
7	25	1.398	77.49	1.889
8	30	1.477	107.04	2.030
9	35	1.544	119.47	2.077
10	40	1.602	131.95	2.120
11	60	1.78	201.10	2.303
12	100	2.000	221.14	2.345

A. Application of Freundlich Model

According to re. [24], Freundlich mentioned that the following experimental equation is suitable for studying the kinetic mechanism of adsorption of ions or molecules on solid surfaces in a liquid medium;

$X = kC_n$

And advised that the logarithmic form of this equation could be used to make it more suitable for application as follows;

$Log\ X = n\ Log\ C + Log\ k$

whereas:

X = the amount of ions or salts, adsorbed on the unit weight of surfaces of the solid body molecules in the liquid phase (meq/100g).

C = concentration of ions or salts in the liquid phase at equilibrium.

n, k = constants.

It is a first-degree linear equation that can be graphically expressed by applying it on the data of table 2. As it appears from this table that the relationship between KCl salts adsorbed on the surfaces of the solid phase of the biochar particles, expressed as (Log X), and the KCl salts dissolved in the liquid phase expressed as (Log C), is a first degree relationship on the form of a straight line sloped by the constant (n) and intersects the axis of (Log X) at the point below zero by the constant (Log K), as shown in Figure (1).

$$\text{Log}\mathbf{X} = 1.868\text{Log}\mathbf{C} - 0.677$$
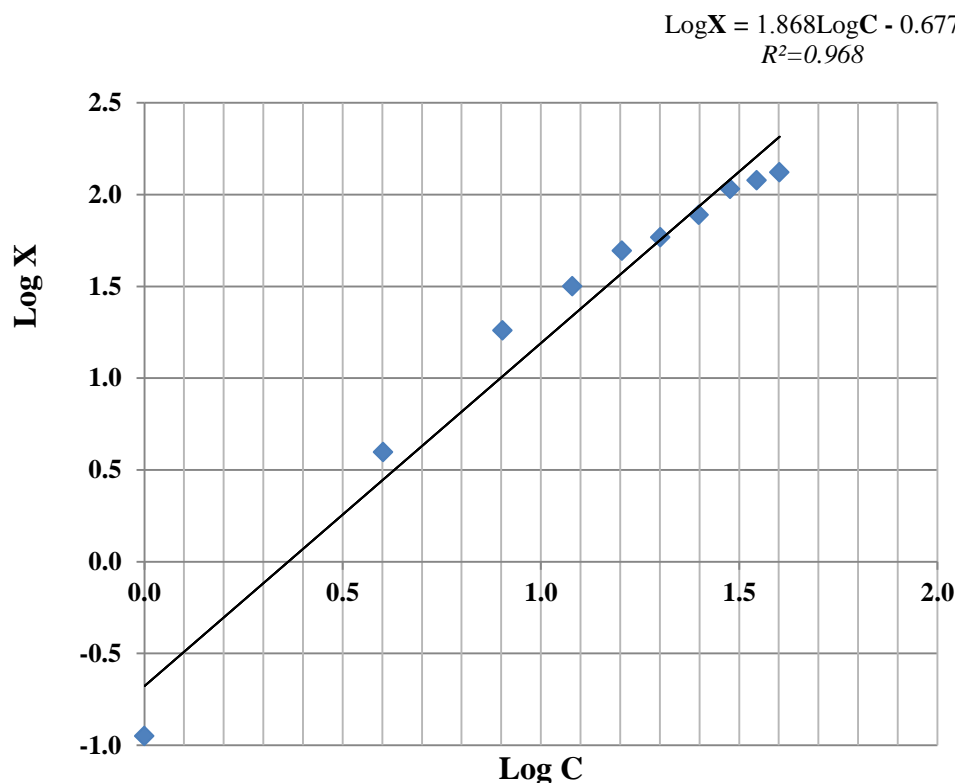$$R^2 = 0.968$$

Figure (1): The relationship between the logarithmic values of soluble KCl concentrations in the liquid phase, (LogC), and the logarithmic values of the adsorbed KCl concentrations on the surfaces of biochar particles in the solid phase, (LogX), at the equilibrium stage according to Freundlich equation.

Figure (1) shows that there is a strong relationship between the two variables (LogC) and (LogX), where (R = 0.984) and (R2 = 0.968) in consistent with the finding of [25] with NH4+ and NO3- adsorption by deferent kinds of biochars, noting that the negative value of the straight line intersection with the (LogX) axis (b = -0.677) recorded in the case of the blank when no KCl salt was added to the medium in the blank treatment (C=0), caused by the presence of some remains of soluble salts [26], held into the micro-pores of the biochar particles, that contributed in starting the descriptive straight line of this relationship from negative point .

Also, this relationship line, does not guarantee its validity if used values are outside the tested points, especially outside the straight-line range of the first ten tested points.

Therefore, the Freundlich equation cannot be used to predict the saturation limit or the maximum adsorption limit at the top of the graph curve according to [27].

B. Applying a First Order Equation :

Experimenting a hypothetical first order equation (X = kC + b), by applying it on the variables of table (2) to verify the kind of the relationship between the concentrations of dissolved salts in the liquid and adsorbed salts on the sites of the solid phase surfaces, expressed in the two dependent variables (C) (meq/L) and (X) (meq/100g), through the representative line of this relationship at the equilibrium stage between the liquid and solid phases, where (k) and (b) are constants expressing the slope of the straight line and its intersection point with the (X) axis, where the square correlation coefficient (R²) recorded a high value (0.996), Up to the point of salt concentration of (60 meq/L)in the liquid phase, meanwhile, at concentrations above this point the relationship begins to decline and shift towards a second degree order equation in which the value of (R²) decreases as a result of the beginning of the straight line turning into a curved line, Figure (2).

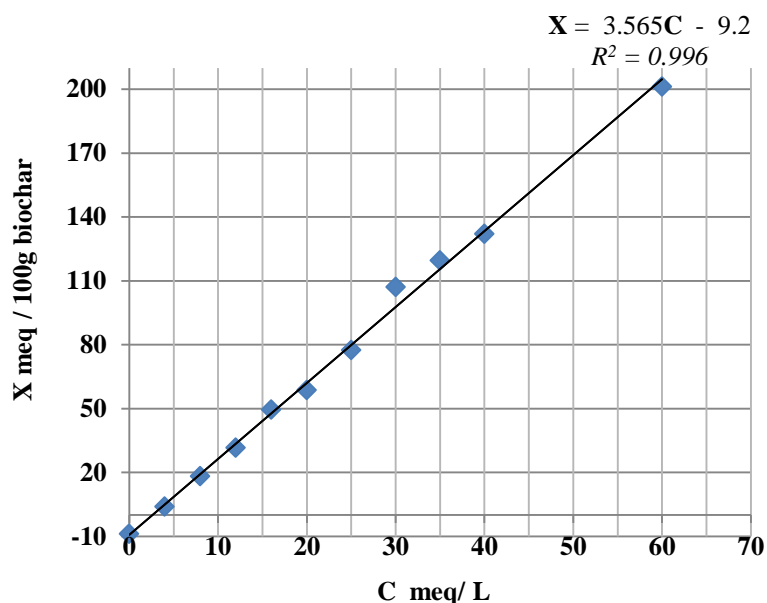$$\mathbf{X} = 3.565\mathbf{C} - 9.2$$
$$R^2 = 0.996$$

Figure (2): The relationship between the soluble KCl concentrations in the liquid phase (C) and the adsorbed KCl concentrations on the surfaces of biochar particles in the solid phase (X), meq/100g at equilibrium.

This equation helps in calculating the amount of salt adsorbed on the surfaces of the biochar particles under any concentration of soluble salts in the liquid phase within the limits of the experimental points. Noting that the negative value (b = -9.2) of the straight line intersection with the (X) axis recorded in the case of the blank treatment, when no KCl salt was added to the liquid phase, means that there are some salts held into the micro-spores of biochar particles contributed to reverse supply to the equilibrium system, although the correlation coefficient for this relationship recorded a high value (R = 0.998) and the correlation coefficient square also recorded a value (R^2 = 0.996), which reflex the accuracy of this relationship. It is assumed that the limits of the quantities of KCl salt adsorbed on the surfaces of the solid phase are determined by the constant (b) as a minimum and maximum limits, but it appears that the latter can only be determined at the point at which the straight line begins to decline, or shift towards a curved shape, which cannot be achieved in the application of this straight-line equation, which maintains its straightness at the equilibrium points with increasing salt concentrations in the liquid phase until approaching the point of the saturation of the adsorption surfaces and turning into a second order mathematical relationship between the two variables. This confirms what [28] referred to, in applying the linear Langmuir equation to zinc adsorption in calcareous soils, as it was suitable for characterizing cases at low concentrations only and within the limits of experimental points. As for [29], found different results in variable conditions for different soils with the clay content, (pH) and (CEC), and the solid phase heterogeneity, as the Freundlich equation was unique in characterizing the kinetics of the absorption of potassium. While re. [30] found a good applicability for Freundlich adsorption isotherm under limited ranges of B concentration between (0 to 100 ppm) only, for 10 different soils.

C. Second Order Equation :

The application of the second order exponential equation to the logarithmic values of the soluble salt concentrations of KCl added to the liquid phase in the medium (C), and adsorbed on the surfaces of the biochar particles in the solid phase (X), in the same medium, as shown in Table (1), Whereas, the relationship between LogC and LogX in this equation appears as a curved graphical line expressing this relationship, represented by the following equation:

$X = aC2 + bC + k$

By replacing the symbols of the equation with the logarithmic values of (C) and (X), according to Table 2, the equation becomes in its final form as follows:

$LogX = a (LogC)2 + b LogC + k$

By replacing the constants of the equation (a, b, k) with the actual values according to the relationship graph between (C) and (X), for the experimental points in Table 2 and Figure 3, the equation becomes as follow:

$LogX = - 0.669 (LogC)2 + 2.992 LogC - 0.948$

whereas:

Log X = the logarithmic values of the adsorbed KCl salts concentrations on solid phase surfaces, meq/100g biochar.

LogC = logarithmic values of the dissolved concentrations of KCl salts in the liquid phase, meq/L .

Figure (3) shows the tendency of the relationship between these two variables (LogC) and (LogX), in the form of a graphical curve, in which, the rate of increase in the adsorbed salts on the surfaces of the biochar particles seems to decrease as it approaches the saturation limit of the adsorption surfaces.

Also, the value of (R2 = 0.999), and therefore the high value of (R = 0.9995), indicates a very strong and accurate correlation between LogC and LogX variables.

$$LogX = -0.669(LogC)^2 + 2.992LogC - 0.948$$
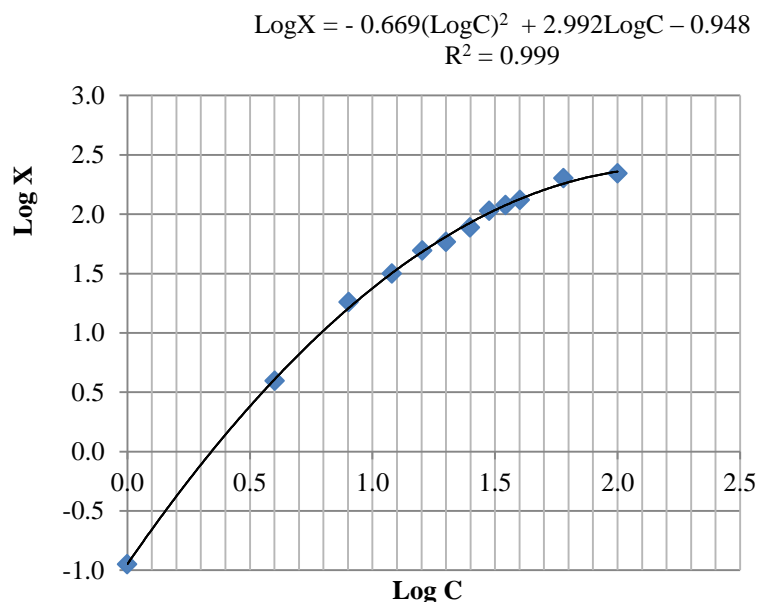$$R^2 = 0.999$$

Figure (3): The relationship between the logarithmic values of the soluble KCl concentrations in the liquid phase (logC), and the adsorbed KCl concentrations on the surfaces of biochar particles in the solid phase (logX), meq/100g, at equilibrium.

Noting that the expression of this relationship through the application of a second-order mathematical equation makes it distinguished than the other two equations,(Freundlich and the previous first-degree hypothetical equation) by allowing to predict any new default values for the adsorbed salts that can be reached when adding new concentrations of dissolved salts to the liquid phase outside the experimental limits, especially to predict the maximum adsorption level, using the inverse logarithm of the obtained predicted values (e), Whereas if: logX = e then: X = 10e

IV. CONCLUSION

Adsorption of KCl salt on the adsorption sites of the surfaces of biochar particle sprepared from olive mill residues, as a solid phase, is strongly correlated with the increase in the salt concentrations in the liquid phase at equilibrium in the system of (KCl/BioC).The kinetic isotherm of this equilibrium, strongly follows first degree hypothetical equation as (R2 = 0.996) which is closely related to the logarithmic equation of Freundlich (R2 = 0.968), within concentrations of KCl in the liquid phase below the saturation limit of the adsorption surfaces. Which means that the adsorption surfaces of this type of biochar particles, are not characterized by the complete homogeneity of the sites of the adsorption

surfaces as a result of the containment of this biochar to some impurities, especially a small percentage of non-charred organic carbon (4.24%), which gives additional adsorption sites that are not stable in the equilibrium system, creating heterogeneity in the adsorption capacity of some sites on the surfaces of the solid phase particles.

As for, the application of these two equations in the first and second models on the experimental points of KCl concentrations in the liquid phase equivalent or higher than 60 meq/liter, then the application of the second order hypothetical equation becomes more appropriate because it is more expressive and more accurate as it is in the third model, where the square of the correlation coefficient recorded high accuracy (R2 = 0.999), and the resulting curved line equation allows prediction of any hypothetical adsorption value related to the dissolved concentration in the liquid phase when the adsorption surfaces are totally saturated with KCl salt.

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