Mechanism of Copper (II) Removal from Aqueous Solution Using Activated Carbon Prepared from Different Agricultural Materials

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Abstract— Uptake of copper (II) from aqueous solution using activated carbons prepared from nipa palm nut (NPN), palmyra palm nut (PPN), oil palm empty fruit bunch (EFB), oil palm fiber (OPF), and oil palm shell (OPS) was studied. Chemical activation method was adopted in preparing the activated carbon. The effects of various parameters such as pH, adsorbent dosage, contact time, initial ion concentration and adsorbent particle size were investigated in order to determine the adsorption performance of the adsorbents. It was found that the amount of Cu$^{2+}$ adsorbed increased with increase in adsorbent dosage, and contact time and decreased with increase in particle size and initial ion concentration. The optimum pH of 6 was obtained for all the adsorbents. The result of adsorption studies showed that activated carbons produced from NPN and PPN are the most efficient adsorbents for the removal of Cu$^{2+}$ from aqueous solutions while EFB, OPF and OPS were found to be poor adsorbents for the removal of the adsorbate. Adsorption data were fitted to Langmuir, Freundlich, Temkin and Dubinin-Radushkevich equations. Equilibrium data fitted well to the Freundlich, Langmuir and Tempkin models. The equilibrium data were best described by Freundlich model.

Keywords— Adsorption, Copper, Adsorbents, Isotherm, Removal and Activated Carbon

I. INTRODUCTION

The progressive increase of industrial technology result in continuous increase of pollution, so that a great effort has been devoted for minimizing these hazardous pollutants and therefore avoiding their dangerous effects on animals, plants and humans (Al-Omair and El- Sharkawy, 2001). Heavy metal ions are reported as priority pollutants, due to their mobility in natural water ecosystems and due to their toxicity (Volesky and Holan, 1995). These heavy metals are not biodegradable and their presence in streams and lakes leads to bioaccumulation in living organisms causing health problems in animals, plants, and human beings (Ong et al, 2007). Since copper is a widely used material, there are many actual or potential sources of copper pollution. Copper is essential to life and health but, like all heavy metals, is potentially toxic as well. For example, continued inhalation of copper-containing spray is linked with an increase in lung cancer among exposed workers.

Several methods such as ion exchange, solvent extraction, reverse osmosis, precipitation, and adsorption have been proposed for the treatment of wastewater contaminated with heavy metals (Gupta, 2003) Among several chemical and physical methods, the adsorption onto activated carbon has been found to be superior to other techniques in water-re-use methodology because of its capability for adsorbing a broad range of different types of adsorbates efficiently, and its simplicity of design (Ahmad et al, 2006).

However, commercially available activated carbons are still considered expensive (Chakraborty et al, 2005). Consequently, many researchers have studied cheaper substitutes, which are relatively inexpensive, and are at the same time endowed with reasonable adsorptive capacity. In recent years, the preparation of activated carbons from several agricultural by products has been emphasized due to the growing interest in low cost activated carbons from renewable biomass, especially for applications concerning treatment of drinking water and wastewater (Castro et al, 2000). Several works have been carried out on the preparation of activated carbons from sawdust, ficus religiosa leaves, dead biomass, coconut shell, rice husk, and nypa fruticans shoot (Ahmad et al, 2008; Qaiser et al, 2007; Geu et al, 2006; Sekar et al, 2004; Ong et al, 2007; Wankasi et al, 2004). The present study is aimed at the preparation of activated carbon from five different agricultural residues and comparing their performance for the removal of Cu$^{2+}$ from aqueous solution.

II. MATERIALS AND METHODS

A. Sourcing and Pre-treatment of Raw Materials

The raw materials used for the preparation of activated carbon are oil palm shell (OPS), oil palm fiber (OPF), oil palm empty fruit bunch (EFB), nipal palm nut (NPN) and palmyra palm nut (PPN). Oil palm shell, oil palm fiber and oil palm empty fruit bunch were obtained from Ichida women cooperative oil processing mill, Ichida, Anambra State, Nigeria. Nipa palm nuts were obtained from a mangrove in Nkpogu village, Rivers State while Palmyra palm nuts were obtained from Nnamdi Azikiwe University, Awka, Anambra State, Nigeria. The raw materials were washed several times using de-ionized water to remove all traces of impurities, oil, dirt, dust, etc. The materials were dried in the sun for 72 hours to remove excess water until constant weight was obtained.

B. Production of Activated Carbon

The washed and dried raw materials were ground into fine particles using a Jencod grinding machine and sieved to a
particle size of 300μm. 200g of sample was impregnated with concentrated orthophosphoric acid at the acid/precursor ratio of 0.4:1 (wt basis) for OPS and 2:1 for other materials. The impregnated samples were dried in an oven at 120°C for 24hrs. The dried samples were carbonized in a Muffle furnace for 1hr at 500°C for NPN, 2hrs at 500°C for PPN and 2hrs at 800°C for EFB, OPF and OPS. After cooling to the ambient temperature, the samples were washed with de-ionized water several times until pH 6-7, filtered with Whatman No.1 filter paper and then dried in the oven at 110°C for 8hours. The samples were crushed and passed through different sieve sizes and then stored in a tight bottle ready for use.

**C. Characterization of Activated Carbon**

The pH of the carbon was determined using standard test of ASTM D 3838-80 (ASTM, 1996). Moisture content of activated carbon and raw materials was determined using ASTM D 2867-91 (1991). The bulk density of the activated carbon was determined according to the tamping procedure by Ahmeda et al (1997). The volatile content was determined by weighing 1.0g of sample and placing it in a partially closed crucible of known weight. It was then heated in a muffle furnace at 900°C for 10mins. The percentage fixed carbon was determined as100 – (Moisture content + ash content + volatile matter). The iodine number was determined based on ASTM D 4607-86 (1986) by using the sodium thiosulphate volumetric method. The specific surface area of the activated carbon was estimated using Sear’s method (Al-Qadah and Shawabkah, 2009 and Alzaydien, 2009) by agitating 1.5g of the activated carbon samples in 100ml of diluted hydrochloric acid at a pH = 3. Then a 30g of sodium chloride was added while stirring the suspension and then the volume was made up to 150ml with deionized water. The solution was titrated with 0.1N NaOH to raise the pH from 4 to 9 and the volume, V recorded. The surface area according to this method was calculated as S = 32V – 25. Where, S = surface area of the activated carbon, V = volume of sodium hydroxide required to raise the pH of the sample from 4 to 9.

**D. Adsorbate Preparation and Batch Adsorption Studies**

The reagent used was copper sulphate (CuSO₄) and de-ionized water. The reagents were of high grade. The sample (CuSO₄) was dried in an oven for 2hrs at 105°C to remove moisture. 2.51g of CuSO₄ was dissolved in 1000ml of de-ionized water to get the stock solution of 1000g/l. A predetermined amount of activated carbon was added to 100ml of the 100mg/l of adsorbate in a conical flask and placed on a magnetic stirrer. The stirring was done at different temperature for different times. After adsorption is complete, the solution was filtered using Whatman no.1 filter paper. The residual Cu²⁺ concentration of the effluent was determined spectrophotometrically using atomic adsorption spectrophotometer at 324.8nm wavelength. The adsorption capacity, qₑ (mg/g) and percentage adsorbed were calculated from Eq. (1) and Eq. (2) respectively.

\[
qₑ = [(Cₒ – Cₑ) V / m]
\]

\[
% \text{ Adsorbed} = [(Cₒ – Cₑ) / Cₒ] \times 100
\]

Where, Cₒ and Cₑ are the metal concentrations (mg/l) at initial and any time t, respectively, V the volume of the solution (L), and m the mass of adsorbent used (g). The effect of particle size on the sorption of Pb²⁺ was investigated by changing the particle size in the range of 150 to 1100μm with initial concentration of 100mg/l and adsorbent dosage of 0.5g for 3hrs. Initial ion concentration of 100mg/l was used in conjunction with adsorbent dosage 0.2, 0.35, 0.5, 1.0, 1.5 and 2.0g for contact time of 3hrs and particle size of 150μm. The effect of pH on the sorption was studied at pH 3, 4, 5, 6, 7, 8 with 0.5g of adsorbent dosage, for 3hrs. The effect of contact time was investigated for 10, 20, 40, 60, 90, 120, 150 and 180 min at pH 7, 0.5g/100ml sample dosage and at temperatures of 25-50°C. The effect of initial Cu²⁺ concentration on the sorption was investigated by changing the initial ion concentration in the range of 100-750mg/l with 150μm particle size, adsorption dosage of 0.5g and at 25-50°C for 3hrs.

### III. RESULTS AND DISCUSSION

Physico-chemical characteristics of activated carbons are shown in Table 1. It can seen that OPS had the lowest moisture and ash contents and high surface area of 863m²/g and iodine value of 810.75 mg/g. NPN had the lowest bulk density and fixed carbon, and highest surface area and iodine number. OPF had the lowest iodine number and surface area. The presence of ash has been shown to inhibit surface development (Valix et al, 2004). Consequently low surface area is observed in carbons with high ash content (Pendyal et al, 1999). The raw material, the particle size range used and the degree of activation affect bulk density, which does have an effect on adsorption per unit volume (Yenisoy-Karakas et al, 2004). The iodine number value is an indication to surface area of the activated carbon (Abdullah, 2001). The larger the surface area is the greater is the adsorption capacity.

<table>
<thead>
<tr>
<th>Activated Carbon wt %</th>
<th>Moisture content wt %</th>
<th>Volatile matter wt %</th>
<th>Ash content wt %</th>
<th>Fixed carbon</th>
<th>Bulk Density, g/cm²</th>
<th>pH</th>
<th>Iodine number (mg/g)</th>
<th>Surface area (m²/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>OPF</td>
<td>3.43</td>
<td>23.61</td>
<td>4.39</td>
<td>72.0</td>
<td>0.56</td>
<td>6.6</td>
<td>676.88</td>
<td>715.63</td>
</tr>
<tr>
<td>EFB</td>
<td>3.22</td>
<td>22.42</td>
<td>4.0</td>
<td>73.58</td>
<td>0.58</td>
<td>6.7</td>
<td>802.45</td>
<td>850.11</td>
</tr>
<tr>
<td>OPS</td>
<td>2.15</td>
<td>20.3</td>
<td>3.0</td>
<td>76.67</td>
<td>0.64</td>
<td>6.7</td>
<td>810.75</td>
<td>863.42</td>
</tr>
<tr>
<td>NPN</td>
<td>4.80</td>
<td>24.60</td>
<td>3.88</td>
<td>71.52</td>
<td>0.53</td>
<td>6.9</td>
<td>815.62</td>
<td>871.22</td>
</tr>
<tr>
<td>PPN</td>
<td>4.10</td>
<td>18.14</td>
<td>3.30</td>
<td>78.56</td>
<td>0.61</td>
<td>6.8</td>
<td>785.78</td>
<td>820.37</td>
</tr>
</tbody>
</table>
A. Effect of Adsorption Dosage on the Adsorption Process

Adsorption dosage is an important parameter as it determines the capacity of an adsorbent for a given initial concentration of the adsorbate. The result showed that as the adsorbent dosage increased, the percentage of adsorption also increased but the amount adsorbed per unit mass of the adsorbent decreased considerably (Figure 1). The decrease in unit adsorption with increasing dose of adsorbent is attributed to adsorption sites remaining unsaturated during the adsorption reaction (Bulut and Aydin, 2005). Figure 1 reveals a definite increase in the adsorption capacity of the adsorbents with dosage. This is as a result of the larger number of available adsorption sites favouring the enhanced uptake of the metal ions (Karthikeyan et al., 2008). The authors stated that the dependence of adsorption on doses can be explained in terms of the concentration factor involving low metal-to-adsorbent ratio at low metal concentrations. At higher concentrations, the available sites of adsorption become fewer and hence the percentage of metal ion removed is dependent upon the initial concentration. Many workers obtained similar results (Gueu et al., 2006, Maheswani et al., 2009, Sekar et al., 2004).

B. Effect of Particle Size on the Adsorption Process

The removal of Pb^{2+} at different particle sizes showed that the removal rate increased as particle size deceased (Fig. 2). This same trend was reported by Demirbas et al. (2004) and Sekar et al. (2004). The relatively higher adsorption with smaller adsorbate is explained by the fact that smaller particle yields large surface area. According to Weber and Morris (Karthikeyan et al., 2004), the breaking of larger particles tends to open tiny cracks and channels on the particle surface of the material resulting in more accessibility to better diffusion, owing to the smaller particle size. NPN and PPN gave the best performance in removal of Cu^{2+} from aqueous solution. This may be attributed to their high surface areas.

C. Effect of pH on the Adsorption Process

One of the most factors influencing not only site dissociation, but also the solution chemistry of heavy metal is pH. Figure 3 presents the effect of pH on the removal of Cu^{2+} by the different adsorbents. The adsorption of the Cu^{2+} increased with increasing pH. The maximum Cu^{2+} adsorption for all the adsorbents was obtained at pH 7. Cu^{2+} adsorption decreased as pH rose beyond the optimum pH of 7. The increase in adsorption capacity of Cu (II) with increasing pH is attributable to two possible mechanisms, which are cation exchange and surface complexation mechanisms.

D. Effect of Initial Ion Concentration on the Adsorption Process

The initial metal concentration of an effluent is important since a given mass of sorbent can only adsorb a fixed amount of heavy metal. From Fig. 4 it is seen that the adsorption percentage decreased with increase in initial ion concentration. However, the amount of Cu^{2+} adsorbed increased with increase in initial ion concentration.

E. Effect of Contact Time on the Adsorption Process

The result of percentage Cu^{2+} adsorbed is shown in Fig. 5.
F. Isotherm Studies

The equilibrium adsorption isotherm is important in the design of adsorption systems (Bulut and Aydin, 2006). In general, the adsorption isotherm describes how adsorbate interact with adsorbents and this is critical in optimizing the use of adsorbents. The relationship between the amount of a substance adsorbed at constant temperature and its concentration in the equilibrium solution is called the adsorption isotherm.

G. Langmuir Isotherm Model

The Langmuir adsorption model is given by:

\[ q_e = \frac{Q b C_e}{1 + b C_e} \]  

(3)

The Langmuir equation can be described by the linearized form (Sekar et al, 2004; Sivakumar and Palanisamy, 2009).

\[ \frac{C_e}{q_e} = \frac{1}{Q_o b} + \left(\frac{1}{Q_o}\right) C_e \]  

(4)

Where \( C_e \) is the equilibrium concentration of the adsorbate, \( q_e \) the amount of adsorbate per unit mass of adsorbent (mg/g), and \( Q_o \) and \( b \) are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. When \( C/C_e \) is plotted against \( C_e \), straight line with slope \( 1/Q_o \) indicates that adsorption follows the Langmuir isotherm. The Langmuir constants, \( Q_o \) and \( b \) were evaluated from the intercept and the slope of the linear plot of experimental data of \( 1/q_e \) versus \( 1/C_e \) and presented in Table 2 to 6. The essential characteristics of the Langmuir equation can be expressed in terms of a dimensionless separation factor, \( R_L \), defined as (Ahmed et al, 2006).

\[ R_L = \frac{1}{1 + b Q_o} \]  

(5)

Where, \( C_0 \) is the initial ion concentration, \( b \) the Langmuir’s adsorption constant (L/mg). The \( R_L \) value implies the adsorption to be unfavourable (\( R_L > 1 \)), Linear (\( R_L = 1 \)), favourable (\( 0 < R_L < 1 \)), or irreversible (\( R_L = 0 \)) (Maheswari et al, 2008). \( R_L \) values for Cu\(^{2+}\) on the adsorbents were less than 1 and greater than zero indicating favourable adsorption under conditions used in this study. However, the values of \( R_L \) for Pb\(^{2+}\) adsorption on EFB, OPF and OPS range from 0.9259 to 0.9434 (which are close to unity) indicating not too favourable adsorption. The correlation coefficients showed that Pb\(^{2+}\) adsorption on adsorbents follow Langmuir. Some researchers showed that experimental data conformed to Langmuir model (Sahmoune et al, 2008; Sekar et al, 2004 and Goyal et al, 2008).

H. Freundlich Isotherm Model

The Freundlich adsorption isotherm can be written as (Lin and Juang):

It was found that the Cu\(^{2+}\) adsorption percentage increased with increasing contact time. Above 90% of Cu\(^{2+}\) adsorption occurred in the first 10 to 60mins for NPN and PPN and thereafter the rate of adsorption of the adsorbate species into the adsorbent was found to be slow. Adsorption equilibrium was reached at 90, 90, 120, 150, and 150mins for NPN, PPN, EFB, OPF and OPS respectively. The initial rapid adsorption is due to the availability of the positively charged surface of the adsorbents for adsorption of anionic Cu\(^{2+}\) species present in the solution. The later slow adsorption is probably due to the electrostatic hindrances between adsorbed negatively charged adsorbate species into the surface of adsorbents and the available anionic adsorbate species in solution, and the slow pore diffusion of the solute ion into the bulk of the adsorbent (Goswani and Ghosh, 2005).
\[ q_e = K_F C_e^{1/n} \]  \hspace{1cm} (6)

A linear form of this expression is:

\[ \log q_e = \log K_F + 1/n \log C_e \]  \hspace{1cm} (7)

Where \( K_F \) and \( n \) are Freundlich constants, \( n \) giving an indication of how favourable the adsorption process is and \( K_F \) is the adsorption capacity of the adsorbent. \( K_F \) and \( n \) are determined from the linear plot of \( \log q_e \) versus \( \log C_e \). From the linear plots of \( \log q_e \) versus \( \log C_e \), values of Freundlich constants \( K_F \) and \( n \) were calculated from the intercept and slope, respectively, and are presented in Tables 2 to 6. The \( K_F \) which is a measure of adsorption capacity, decreased with increased temperature for \( Cu^{2+} \). The correlation coefficients were greater than 0.904. The exponent is 1\( < n < 3 \), showing beneficial adsorption for the system. The correlation coefficients, \( R^2 \) ranged from 0.978 to 1 indicating that \( Cu^{2+} \) adsorption followed Freundlich model. Similar results are obtained by Aksu and Tezer (2000), Maheswari et al (2008), Sivakumar and Palanisamy (2009) and Goswami and Ghosh (2005).

I. Temkin Isotherm

The Temkin isotherm has been used in the following form (Choy et al, 1999):

\[ q_e = \frac{RT}{b} \ln(A C_e) \]  \hspace{1cm} (8)

A linear form of the Temkin isotherm can be expressed as:

\[ q_e = \frac{RT}{b} \ln A + \frac{RT}{b} \ln C_e \]  \hspace{1cm} (9)

Where \( RT/b = B \) and \( A \) is Temkin constant, \( 1/g. \) The adsorption data can be analyzed according to equation 9 and the constant \( B \) determines the heat of adsorption. The values of Temkin constants \( A \) and \( b \) as well as the correlation coefficients are listed in Tables 2 to 6. All correlation coefficients were greater than 0.904. Generally, data fairly conformed to Temkin isotherm model. The values of \( A \) decreased with increase in temperature while the values of \( b \) increased with increase in temperature for \( Cu^{2+} \) adsorption. The values of constants \( A \) and \( b \) obtained in this work are similar to the values obtained by Sekar et al (2004).

J. Dubinin-Radushkevich Isotherm Model

The Dubinin – Radushkevich equation has the following form:

\[ q_e = q_m e^{-\beta \varepsilon^2} \]  \hspace{1cm} (10)

A linear form of Dubinin – Radushkevich equation isotherm (Sivakumar and Palanisamy, 2009) is:

\[ \ln q_e = \ln q_m - \beta \varepsilon^2 \]  \hspace{1cm} (11)

Where \( q_m \) is the Dubinin– Radushkevich monolayer capacity ( \( mmol / g \)), \( \beta \) a constant related to sorption energy, and \( \varepsilon \) is the Polanyi potential which is related to the equilibrium concentration as follows:

\[ \varepsilon = RT \ln \left(1 + \frac{1}{C_e} \right) \]  \hspace{1cm} (12)

Where, \( R \) is the gas constant (8.314/J/molK) and \( T \) is the absolute temperature. The constant \( \beta \) gives the mean free energy, \( E \), of sorption per molecule of the sorbate when it is transferred to the surface of the solid from infinity in the solution and can be computed using relationship (Lin and Juang, 2002).

\[ E = \frac{1}{\sqrt{2\beta}} \]  \hspace{1cm} (13)

The Dubinin-Radushkevich constants were evaluated and given in Tables 2 to 6. The correlation coefficients ranging from 0.500 to 0.889, indicates that the adsorption of \( Cu^{2+} \) on NPN and PPN did not follow the Dubinin-Radushkevich isotherm. However, adsorption of \( Cu^{2+} \) on EFB, OPFand OPS conform to Dubinin-Radushkevich isotherm. If the mean free energy, \( E \) is \( <8KJ/mol \), the adsorption is physisorption and if the energy of activation is 8-16KJ/mol, the adsorption is chemisorptions in nature (Sarin et al, 2006; Sivakumar and Palanisamy, 2009). Results of \( E \) obtained indicate that adsorption of \( Cu^{2+} \) on NPN and PPN was physisorption while the adsorption of \( Cu^{2+} \) on EFB, OPF and OPS was chemisorptions. B values obtained in this work are similar to the values obtained by Sivakumar and Palanisamy (2009).

### Table 2: Calculated isotherm parameters for the adsorption of \( Cu^{2+} \) on NPN

<table>
<thead>
<tr>
<th>Isotherm model</th>
<th>Temperature, K</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>298</td>
</tr>
<tr>
<td>Langmuir</td>
<td></td>
</tr>
<tr>
<td>( Q ) (mg/g)</td>
<td>166.67</td>
</tr>
<tr>
<td>( b ) (L/mg)</td>
<td>0.00598</td>
</tr>
<tr>
<td>( R^2 )</td>
<td>0.6297</td>
</tr>
<tr>
<td>Temkin</td>
<td></td>
</tr>
<tr>
<td>( b ) (L/mg)</td>
<td>71.03</td>
</tr>
<tr>
<td>( A ) (L/g)</td>
<td>0.062</td>
</tr>
<tr>
<td>( R^2 )</td>
<td>0.946</td>
</tr>
<tr>
<td>Freundlich</td>
<td></td>
</tr>
<tr>
<td>( N )</td>
<td>1.7182</td>
</tr>
<tr>
<td>( K_F ) (L/g)</td>
<td>4.000</td>
</tr>
<tr>
<td>( R^2 )</td>
<td>0.993</td>
</tr>
<tr>
<td>Dubinin-Radushkevich</td>
<td></td>
</tr>
<tr>
<td>( q_0 ) (mg/g)</td>
<td>79.679</td>
</tr>
<tr>
<td>( \beta ) (mol^2/J^2)</td>
<td>0.000</td>
</tr>
<tr>
<td>( E ) (kJ/mol)</td>
<td>0.000</td>
</tr>
<tr>
<td>( R^2 )</td>
<td>0.805</td>
</tr>
</tbody>
</table>
The study shows that the adsorption of Cu²⁺ from aqueous solutions was studied. The potential of NPN, PPN, EFB, OPF and OPS as low-cost adsorbents for the removal of Cu²⁺ from aqueous solutions was studied. The study shows that NPN and PPN are very efficient adsorbents for removing Cu²⁺ from aqueous solutions while EFB, OPF and OPS are very adsorbents for the removal of Cu²⁺ from aqueous solutions. The ranking of adsorbents for Cu²⁺ adsorption showed this order: NPN>PPN>EFB>OPF>OPS. Copper (II) adsorption was found to vary with adsorbent dosage, pH, initial ion concentration, particle size, and contact time. Isotherms for the adsorption of lead on the adsorbents were developed and found to vary with adsorbent dosage, pH, initial ion concentration, particle size, and contact time.

### IV. CONCLUSION

The potential of NPN, PPN, EFB, OPF and OPS as low-cost adsorbents for the removal of Cu²⁺ from aqueous solutions was studied. The study shows that NPN and PPN are very efficient adsorbents for removing Cu²⁺ from aqueous solutions while EFB, OPF and OPS are very adsorbents for the removal of Cu²⁺ from aqueous solutions. The ranking of adsorbents for Cu²⁺ adsorption showed this order: NPN>PPN>EFB>OPF>OPS. Copper (II) adsorption was found to vary with adsorbent dosage, pH, initial ion concentration, particle size, and contact time. Isotherms for the adsorption of lead on the adsorbents were developed and found to vary with adsorbent dosage, pH, initial ion concentration, particle size, and contact time.
the equilibrium data fitted well to the Freundlich, Langmuir and Temkin models. The equilibrium data was best described by Freundlich model.

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