PERIÓDICO TCHÊ QUÍMICA

ARTIGO ORIGINAL

AVALIAÇÃO EXPRESSA DO MECANISMO DE SORÇÃO EM MATERIAIS QUE CONTÊM TURFA

EXPRESS EVALUATION OF SORPTION MECHANISM ON PEAT CONTAINING MATERIALS

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RESUMO

A turfa é um sorvente eficaz de metais pesados devido à sua alta capacidade de troca catiônica. O uso de sorventes de turfa tem muitas vantagens. É um adsorvente barato, acessível e eficaz para uma ampla gama de poluentes ambientais. Além disso, pode ser usado separadamente ou como um absorvente combinado e constituintes complexos; no entanto, também possui várias desvantagens, como resistência mecânica insuficiente e baixa estabilidade química das partículas de turfa. Para superar essas deficiências, vários métodos de modificação de turfa foram desenvolvidos (por exemplo, turfa modificada com quitosana, que é usada como um absorvente para remoção de metais pesados, como turfa). As propriedades de sorção por troca iônica de sorventes a base de turfa e turfa modificada com quitosana foram avaliadas pelo método do medidor de pH. Uma amostra de turfa ativada por ácido também foi investigada. Os experimentos foram realizados em modo estático, utilizando amostras de turfa natural colhidas no lago Sevan (próximo à cidade de Vardenis, na região de Gegharkunik, na República da Armênia). Também foi realizado um estudo semelhante sobre a resina de troca catiônica KU-2-8. O objetivo deste artigo foi a avaliação expressa do mecanismo de sorção de sorventes de turfa. O trocador de cátions foi transferido para a forma H⁺ por tratamento com HCl (1N). Alterações no perfil de pH em função do tempo de exposição indicam sua identidade com o padrão da alteração de pH do trocador de cátions. Verificou-se que a ativação ácida da turfa (exposição de uma amostra de turfa em HCl 1N por 10-16 horas) aumenta sua capacidade de adsorção. Nesse caso, a diminuição máxima no pH é pH = 2,33, 1,44 pontos mais baixo que na turfa (pH = 3,77) e 0,95 pontos mais que no cationita KU-2-8. Isso indica que os sorventes à base de turfa podem ser regenerados. A turfa modificada com quitosana apresentou deterioração na capacidade de sorção. Nesse caso, pH min = 3,9, o que mostra que a sorção na quitosana não é de troca iônica por natureza.

Palavras-chave: turfa, trocador de cátions, sorvente, sorção, mecanismo de sorção.

ABSTRACT

Peat is an effective sorbent of heavy metals, which is due to its high cation-exchange ability. The use of peat sorbents has many advantages. It is an inexpensive, affordable, and effective sorbent for a wide range of environmental pollutants. Also, it can be used either separately or as a combined sorbent and complex constituents; however, it also has several disadvantages, such as insufficient mechanical strength and low chemical stability of peat particles. To overcome these shortcomings, various methods of peat modification have been developed (e.g., peat modified with chitosan, which is used as a sorbent for heavy metals removal like peat). The ion-exchange sorption properties of sorbents on the base of peat and chitosan - modified peat were evaluated by the method of pH meter. An acid-activated peat sample was also investigated. The experiments were carried out in a static mode using natural peat samples taken from the Lake Sevan (near the city of Vardenis of the Gegharkunik region of the Republic of Armenia). Also, a similar study was conducted on KU-2-8 cation exchange resin. The purpose of this article was the express evaluation of the sorption mechanism of peat sorbents. Cation exchanger was transferred to the H⁺ form by treatment with HCl (1N). Changes in the pH profile as a function of the exposure time indicate their identity with the pattern of the pH change of the cation exchanger. It was found that acid activation of peat (exposure of a peat sample in HCl 1N for 10-16 hours) increases its adsorption capacity. In this case, the maximum decrease in pH is pH = 2.33, which is 1.44 points lower than for peat (pH = 3.77) and 0.95 points more than for KU-2-8 cationite. This indicates that sorbents based on peat can be regenerated. Chitosan-modified peat exhibits deterioration in sorption capacity. In this case, pHmin = 3.9, which shows that sorption on chitosan is not ion-exchange in nature.

Periódico Tchê Química. ISSN 2179-0302. (2020); vol.17 (n°34)
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469

1. INTRODUCTION:

The use of sorbents based on waste and natural materials is a very urgent problem and is of great interest since resource-saving and environmental protection are priority directions in modern science and technology, and the use of waste on an industrial scale is economically valuable (Imaga, Abia, 2015a; Imaga, Abia, 2015b; Ezuma, 2014; Cheng, Yu, Wang, Chen, Guo, 2014).

The effectiveness of the sorbent depends on the presence of a well-developed surface and the presence of active sites on this surface. The efficiency of the adsorption process depends not only on the amount of sorbent but also on the chemical nature of its surface, the presence of one or another type of active centers on the surface (Klimov, Buzaeva, 2011, p. 22-25; Novoselova, Sirotkina, 2008a, p. 251; Lishtvan, Dudarchik, Kovrik, Smychnik, 2007, p. 38-42).

Among the disadvantages of natural materials and wastes used as a source material for the sorption materials, is their heterogeneity not only from a structural point of view but also their surface. As a rule, such materials have different functional surface groups, which ultimately determine the sorption properties of sorbents derived from them. Characterize these groups becomes relevant to understand the sorption mechanism of such systems better. Therefore, the purpose of this article is to develop express and cheap methods for determining the sorption mechanism of sorption materials obtained based on peat (Cochrane, Lu, Gibb, Villaescusa, 2006, p. 198; Novoselova, Sirotkina 2008b, p. 64-77; Kalmykova, Stromvall, Steenari 2008, p. 885).

The presence of peat in the composition of a large number of biologically active compounds and many reactive functional groups causes a growing interest in the development of peat sorbents (Lyngsie, Borggaard, Hansen, 2014; Sen Gupta, Curran, Hasan, Ghosh, 2009, p. 954).

Adsorption is an effective method for removing metal ions from contaminated water. Organic components of peat, in particular humic substances, are primarily responsible for providing active adsorption sites on which various mechanisms bound metals. It is believed that the adsorption of metal on peat occurs through a combination of physical adsorption, ion exchange and complexation (Dada, Adekola, Odebunmi, 2017; Fernandes, Almeida, Menezes, Debacher, Sierra 2007, p. 412–419; Sieczka, Koda, 2016; Poznyak, Shimanskaya, 2012).

Among organo-mineral compounds, the most complex is the complex compounds called chelates. Chelation is considered as an equilibrium reaction between metal ions and functional groups of an organic molecule, in which more than one bond forms between the components (Semendyaeva, Marmulev, Dobrotvorskaya, 2011; Hayrapetyan, Gevorgyan, Hayrapetyan, Bareghamyan, Pirumyan, 2016; Batista *et al.*, 2009; Lishtvan, Dudarchik, Kovrik, Smychnik, 2007).

The adsorption complexes of organic substances with the mineral part of peat can be divided into two types: organic films on minerals, resulting from the adsorption of humic substances, insoluble complexes of humic substances with non-silicate forms of sesquicrate oxides (Fe_2O_3, Al_2O_3). In the formation of insoluble products, the primary mechanism is the adsorption of humic substances. The creation of complexes from mineral colloids and humic substances is possible due to the interaction through cations, primarily through iron ions (Hayrapetyan *et al.*, 2016; McLaughlin, Bartholomew, 2007).

It is believed that the primary mechanism of peat sorption is ion-exchange; however, because on the surface of peat, there are many other functional groups (alcohol, aldehyde, carboxyl, ketone, and phenolic) peat also has pronounced complexing properties. Therefore, along with ion exchange, their complexation is added, as a result of which a high degree of sorption is observed (Gondar, Lopez, Fiol, Antelo, Arce, 2005; McLaughlin, Bartholomew, 2007; Lee, Park, Ahn, Chung, 2015; Sen Gupta, Curran, Hasan, Ghosh, 2009).

This hypothesis is based on the functional groups that exist on the humic acid molecule. However, there are no experimental methods that support this hypothesis. The goal of this article was to confirm this statement empirically.

2. MATERIALS AND METHODS:

The choice of pH-meter as a way to study the processes of ion exchange during the contact of the electrolyte with the test materials is dictated, firstly, because the procedure for measuring pH is effortless and very inexpensive, and secondly, ion

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470

exchange is always accompanied by a change in the pH of the medium. The recording of such changes lies at the heart of the methodology of the experiment.

As a criterion, we selected the study of changes in the pH of the model solution in the presence of a robust cation-exchange resin (KU-2-8). The use of cation exchanger serves as a standard for comparing the results obtained.

As a model solution using a 0.1N solution of NaCl. 1 g of peat is placed in different flasks, and 20, 50, 100, 200, 500 ml of the model solution are added, respectively. The pH of the first solution was measured at the initial time, and then after 10, 20, 30, 60, and 1440 minutes. Similar procedures are carried out with peat treated with 1N HCl solution, and with peat modified with chitosan.

The pH value of 0.1 N NaCl and the initial peat is, respectively; pH (0.1 N NaCl) = 5.85, pH (peat) = 4.17.

Samples of peat are dried in a dryer at a temperature of 1050°C to constant weight, then, after grinding, fractionated, passed through sieves.

Activation with hydrochloric acid: a solution of 1N HCl was prepared, added to the sample, and kept for 24 hours. They were then washed with distilled water until neutral pH and air-dried in a dryer at 1050°C.

Peat modification was carried out according to the procedure given in the work by Gevorgyan and Hayrapetyan (2017).

Initially, peat was dried at room temperature. 90 ml of chitosan solution in citric acid (3 g / l) was added to the mass of peat (about 100 g) and dried at room temperature; the pH of the sorbent was 6-7.

To prevent the humates from leaching, an NH_4OH solution with pH = 8–9 was prepared, and 100 ml of the solution was added to a peat-chitosan sorbent (pH = 8–9): the sorbent was dried at room temperature. A solution consisting of 50 ml of water and 15 ml of formaldehyde was added to the peat-chitosan sorbent and left for 2 hours, and then washed with 1000 ml of distilled water, dried in air, then in a dryer at a temperature of 1050°C.

3. RESULTS AND DISCUSSION:

Based on the results, in Figure 1, a curve of pH change depending on the volume of 0.1 N NaCl and the duration of exposure of a suspension

of natural peat in the amount of one gram are plotted.

From Figure 1, it follows that with the addition of 20 ml of the model solution, a relatively sharp decrease in pH is observed (from 5.85 to 3.77-4.17). And with an increase in the exposure time, the first minimum pH value changes from (from 4.17 to 3.77), which is natural; since it takes some time to reach an equilibrium state. In Figure 1, curve of zero exposure in the range from 10 ml to 30 ml gives a loop. The remaining curves with different exposure times do not differ significantly. For greater clarity, Table 1 shows the data based on which the above curves were constructed.

Figure 2 shows the profile of pH change depending on the volume of 0.1 n NaCl suspensions of natural peat, as well as cation exchanger KU-2-8. In contrast to the above experiments, pH values were measured immediately after each addition of a 0.1N NaCl solution at zero exposure. From Figure 2 that after the addition of 10 ml of solution, a decrease in pH is observed (from 5.85 to 3.8) in the case of peat and with an increase in the volume of 0.1 n NaCl of the solution, a gradual increase in pH is observed. For cation, the pH reaches 1.38. It is essential to note the fact that both curves are very similar to each other, which indicates the identity of the sorption processes of Na^+ ions on both materials. The location of the second curve is almost three pH points lower, which is natural since the ion-exchange capacity of pure cation exchanger is much higher than the ion-exchange capacity of any other materials that do not have special attached functional groups (in the case of cation-exchanger-sulfo-groups).

Table 2 presents the data based on which the above curves were constructed. Besides, pH data is shown depending on the time of exposure of the suspension.

It was a similar picture in the case of using natural peat; however, a decrease in pH to 2.5-2.75 is observed. In the case of natural peat, these values were 3.77-4.17. The difference is: (3.77-2.5) ÷ (4.17-2.75) = 1.27-1.42. This difference indicates that as a result of acid treatment, the active centers (including ion exchange) are released from the adsorbed ions of different metals, which leads to an increase in the adsorption capacity, i.e., more Na^+ ions undergo ion exchange at such centers, as a result of which the model solution becomes more acidic because the number of H^+ ions in the solution increases as a result of ion exchange. The next important difference in the pH profile of activated peat is that the zero curve is identical in appearance to the

Periódico Tchê Química. ISSN 2179-0302. (2020); vol.17 (n°34)
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471

others, it is only located on top of all the curves, which is natural since it is shown above that the curves with longer exposures are located lower than the curves responsible for short exposures.

The observed loop in the figures can be explained by the fact that at zero exposure, several adsorption centers do not have time to "release," which leads to the formation of such a loop.

With an increase in the exposure time (after 10 minutes), it seems that as a result of Na^+ competition, these centers are already free, and the ion exchange process proceeds smoothly.

Figure 3 shows the profile of the pH change depending on the volume of the suspension of acid-activated peat in 0.1 n NaCl solutions. The pH was measured directly after adding each portion of 0.1 N NaCl solutions (no exposure time). There is a decrease to pH 2, 64. A similar picture is observed in the case of the use of natural sorbent, however, while decreasing the pH to 3.8. It can be assumed that the acid treatment of peat increases the sorption capacity of the sorbent.

Compared with Figure 2, it can be stated that the curve of pH dependence on the number of the model solution as compared to the curve for acid-activated peat is intermediate between the curves for activated peat and cationite. By the way, here, the "behavior" of the curves is identical.

When comparing the ion-exchange properties of four types of samples (natural peat, acid-activated peat, peat modified by chitosan and cation exchanger KU-2-8 (Figure 4)), it follows that the pH-change profile for the cation exchanger lies below the peat sample curves, which is understandable.

What is important here is the fact that all curves are visually identical, that is. pH curve for the cation exchanger can act as a standard. This identical behavior of peat and peat-containing materials with a cation exchanger indicates that sorption on these systems has a cation exchange character.

Sorption on chitosan does not carry an ion-exchange mechanism, as compared with samples of untreated and activated peat, the pH decrease is the lowest.

There are no similar studies in the literature, although understanding the sorption mechanism is an important issue for understanding the processes in terms of the selectivity of the interaction of the adsorbate with the adsorbent. Therefore, the creation of a simple, inexpensive way to assess the mechanism of sorption of sorbents is an important task.

The method of the experiment is reduced to comparing the profile of the change in the pH of the solution in the presence of a cation exchanger (as a standard) and the pattern of the difference in the pH of the solution in the presence of samples.

The fact that the electrolyte pH changes (0.1 N NaCl) in the presence of a cation exchanger indicates ionic exchange of Na+ for H+, as a result of which H + accumulates in the solution, which causes a decrease in pH. There is no other explanation for the reduction of the pH of the electrolyte solution, i.e., a decrease in pH indicates ion exchange. On the other hand, when building a profile of change in the pH of the electrolyte, a clear picture of the pH depending on the amount of electrolyte is traced, i.e., the classic cation exchanger has a clearly defined pH change.

It is assumed that if the test samples also have such a pH change profile as the standard (cationite), then it can be assumed that ion exchange takes place, i.e., such systems have an ion-exchange mechanism of sorption.

Figure 5 presents the formula of humic acid. It follows from the formula that humic acid, in addition to other functional groups, also has carboxyl groups, it is assumed that these groups are responsible for ion exchange and these groups provide the cation-exchange mechanism for the sorption of peat and peat-containing materials.

It was noted above that acid activation leads to the curing of the sorption capacity of peat materials, this suggests that after removing sorbed cations from the surface of peat-containing materials as a result of acid treatment, the carboxyl groups become H+ forms, which increases their ion-exchange capacity.

This, in turn, means that peat sorbents can be regenerated with the help of acids, which is typical of classical cation exchangers.

There is practically no research of this kind in the literature; therefore, there are no interpretations of the results in the scientific literature on this subject.

We have developed a simple method for assessing the cation-exchange mechanism of sorbents that can be used for other types of sorbents (not only peat). This method is simple, inexpensive, and express.

4. CONCLUSIONS:

A simple, inexpensive method for

evaluating the sorption mechanism of sorbents was proposed, the essence of which is reduced to the identity of the pH change profiles of these materials with the pH change profile of the cation exchanger, and since they are identical in shape, it is assumed that ion exchange takes place.

Peat materials were used as sorption systems. It was established that the treatment of peat with hydrochloric acid increases its sorption capacity, i.e., active sorption centers as a result of acid treatment are released, which leads to an increase in sorption capacity. On the other hand, after modifying the peat surface with chitosan, its sorption capacity decreases, that is, part of the active ion-exchange centers on the peat surface is screened as a result of chitosan binding on the surface. The decrease in the sorption capacity of such systems indicates that sorption on chitosan is not ion exchange in nature.

A simple, express method has been developed for assessing the ion-exchange mechanism of sorption of sorbents, which can be used to characterize not only peat materials. This method allows you to quickly evaluate the mechanism of sorption without the use of expensive instruments and reagents. Therefore, such studies can be useful and can contribute to the knowledge of the properties of sorbents.

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473

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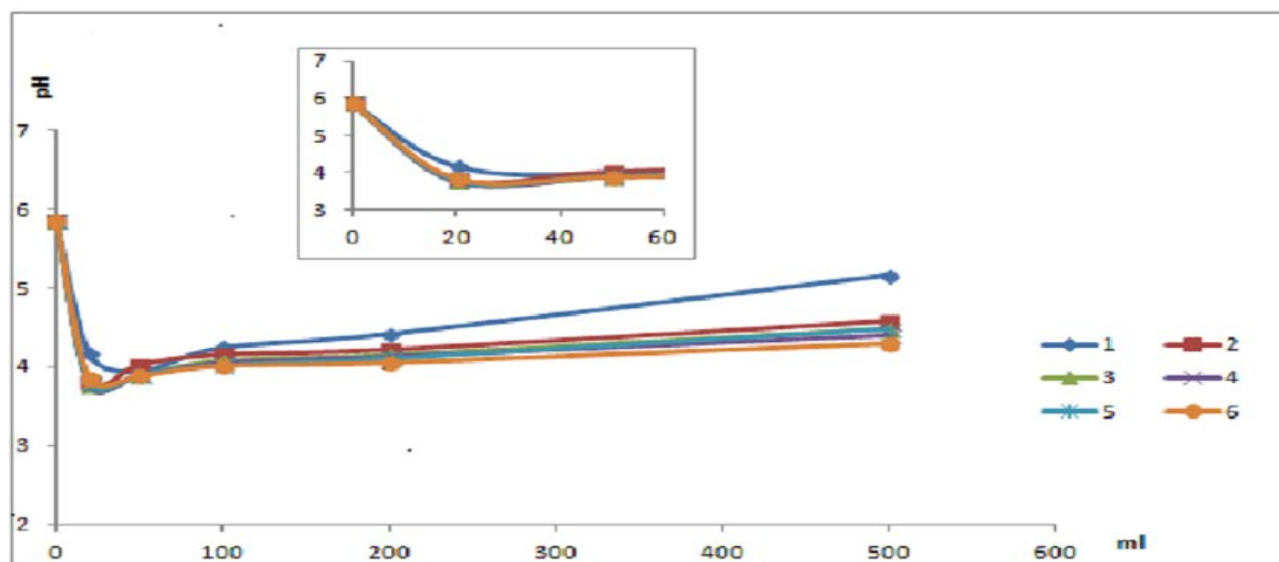40. Yeh, J.T., Chen, C.L., Huang, K.S. *Mater. Lett.,* **2007**, 61, 1292-1295.

Figure 1. *Profile of change in the pH of a suspension of natural peat depending on the amount of a solution of 0.1N NaCl and the time of exposure: 1 - 0 min, 2 - 10 min, 3 - 20 min, 4 - 30 min, 5 – 60 min, 6 - 24 h, amount of peat 1g*

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474

Table 1. *The change in pH of the suspension of natural peat depending on the volume of the solution of 0.1 N NaCl and the exposure time.*

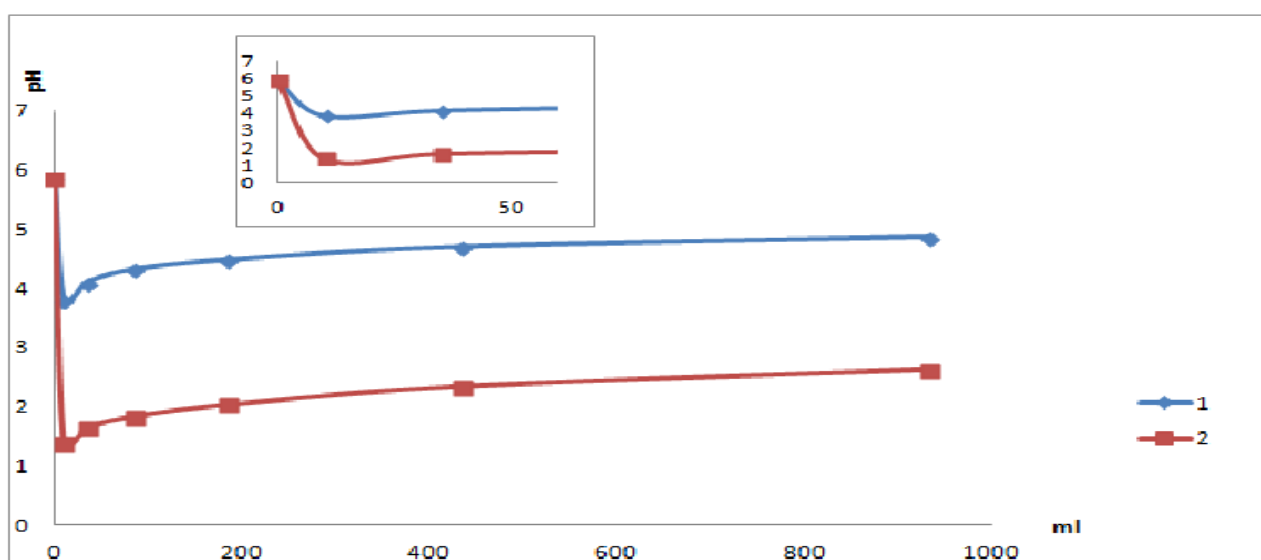| ml	pH_0	$pH_{10minutes}$	$pH_{20minutes}$	$pH_{30minutes}$	$pH_{60minutes}$	$pH_{1440minutes}$
20 ml	4.17	3.81	3.77	3.77	3.79	3.85
50 ml	3.95	4.02	3.89	3.89	3.90	3.89
100 ml	4.25	4.15	4.08	4.05	4.02	4.02
200 ml	4.42	4.21	4.15	4.13	4.11	4.06
500 ml	5.16	4.57	4.48	4.40	4.48	4.30

Figure 2. *Profile of change in the pH of a suspension of natural peat depending on the amount of a solution of 0,1N NaCl compared to cation exchanger: 1 - Natural peat; 2 - KU-2-8 cation exchanger.*

Table 2. *The change in pH of the suspension of peat activated with acid depending on the volume of the solution of 0.1 N NaCl and the exposure time.*

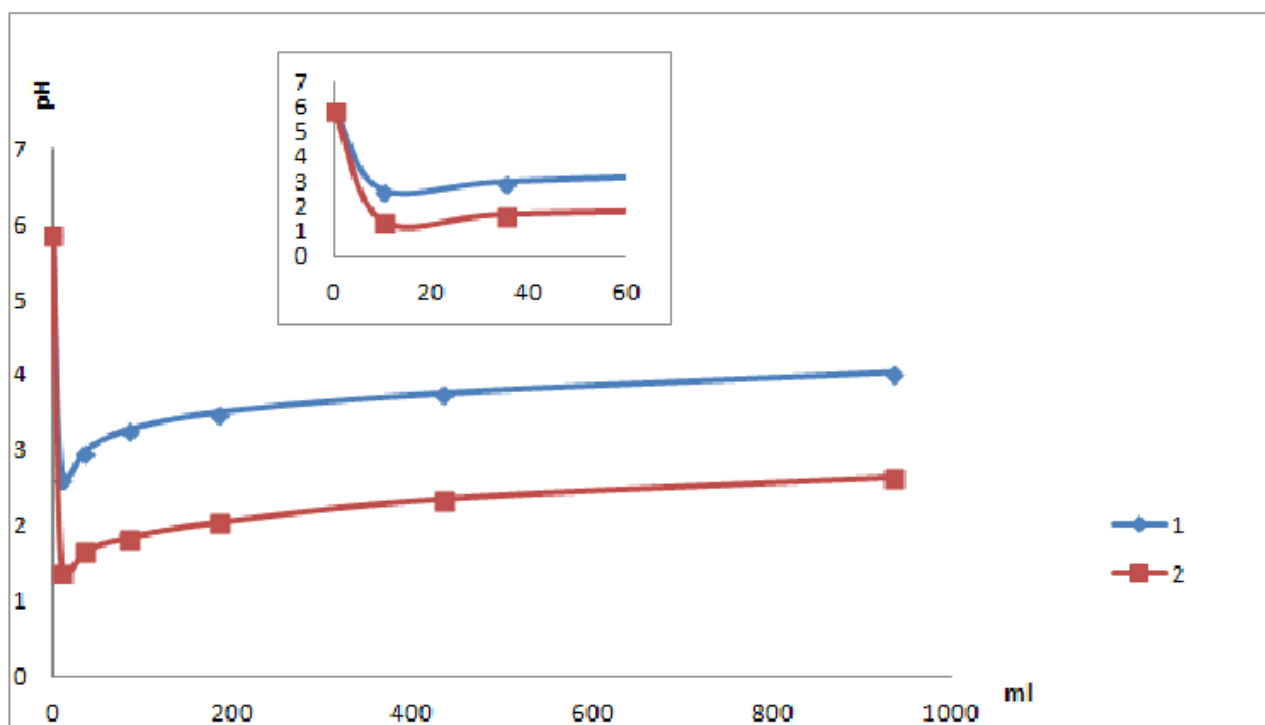| ml	pH_0	$pH_{10minutes}$	$pH_{20minutes}$	$pH_{30minutes}$	$pH_{60minutes}$	$pH_{1440minutes}$
20 ml	2,75	2,50	2,33	2,48	2,47	2,51
50 ml	3,06	2,83	2,78	2,75	2,74	2,76
100 ml	3,32	3,08	2,95	2,92	2,90	2,92
200 ml	3,60	3,27	3,18	3,15	3,12	3,10
500 ml	3,82	3,57	3,49	3,45	3,43	3,41
1000 ml	4,21	3,80	3,72	3,68	3,66	3,60

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475

Figure 3. *Profile of change in the pH of a suspension of peat activated with acid depending on the amount of a solution of 0.1N NaCl compared to cation exchanger: 1 - Natural peat; 2 - KU-2-8 cation exchanger*

Table 3. *The change in pH of the suspension of peat modified with chitosan depending on the volume of the solution of 0,1 N NaCl and the exposure time*

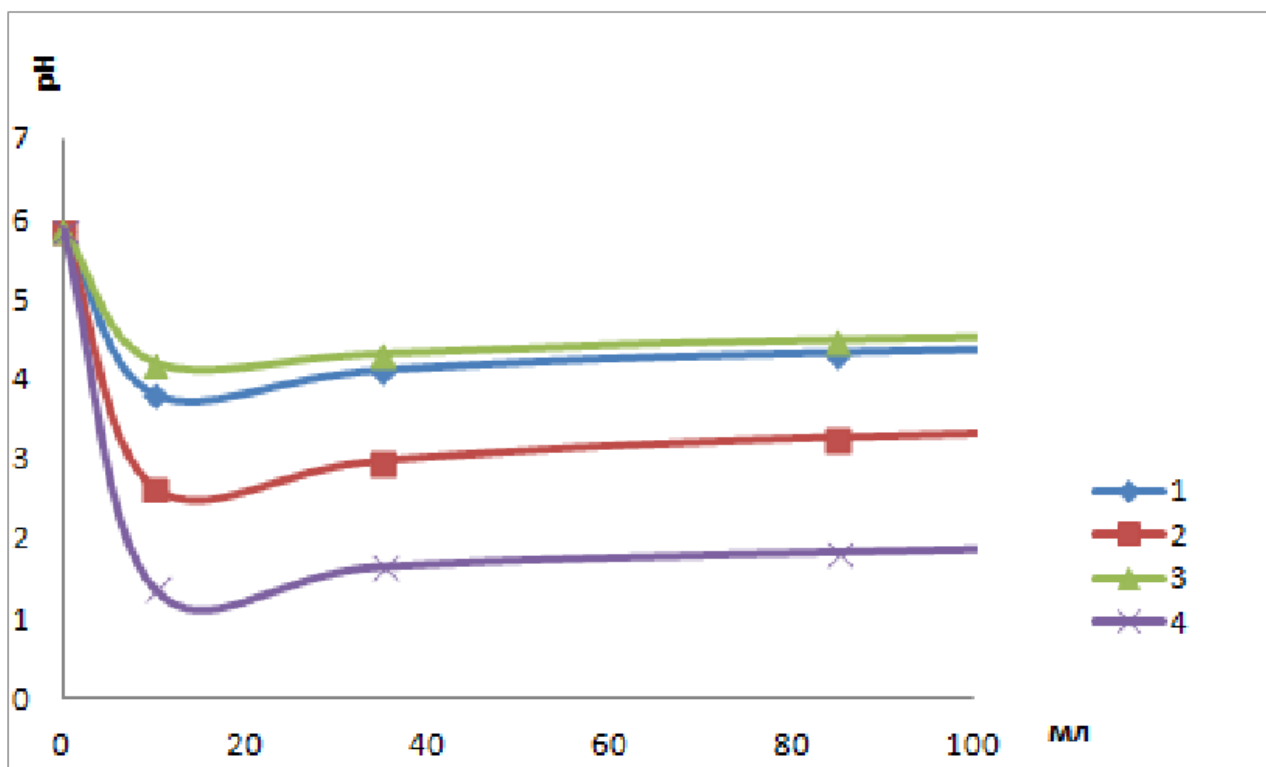| ml	pH_0	$pH_{10мин}$	$pH_{20мин}$	$pH_{30мин}$	$pH_{60мин}$	$pH_{1440мин}$
20 ml	4,00	3,83	3,80	3,80	3,77	3,77
50 ml	4,15	3,90	3,84	3,82	3,79	3,79
100 ml	4,33	4,14	4,11	4,08	4,06	4,06
200 ml	4,60	4,29	4,17	4,16	4,11	4,10
500 ml	4,91	4,53	4,43	4,39	4,33	4,31
1000 ml	5,13	4,73	4,64	4,59	4,54	4,54

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476

Figure 4. *The profile of changes in the pH of a suspension: 1 - Natural peat; 2 - Peat activated with acid; 3 - Modified by chitosan; 4 - Cation exchanger KU-2-8*
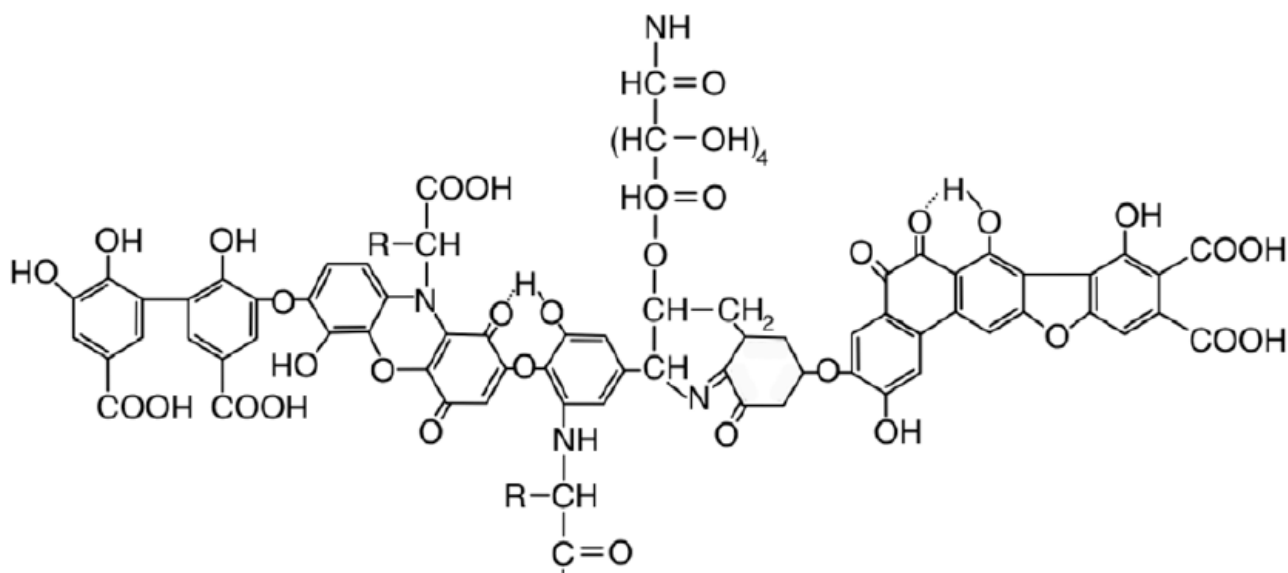

Figure 5. *The formula of humic acid (Stevenson, 1982)*

Periódico Tchê Química. ISSN 2179-0302. (2020); vol.17 (n°34)
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477