



A New Material Based on Clay and its Successful Application to Fix the Glucose

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ABSTRACT

The aim of the present study is to set a new material based on clay and its application to fix the glucose. Montmorillonite KSF-Fe²⁺ were prepared by varying the exchange time (3h, 8h, and 11h). These samples were identified as FM₃, FM₈, and FM₁₁, respectively. Glucose level was determined using the enzymatic reaction. The adsorption kinetic of glucose, by the studied samples, showed that the FM₈ gives a better fixation of glucose (40,5%). Characteristics of the samples were studied by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (IR), thermogravimetry (ATG) and Mossbauer spectroscopy. The X-ray diffraction (XRD) afforded the slight evolution of the interlayer distance d_{001} during cation exchange. The IR spectrum of FM₈-G (Material loaded with glucose) show appearance of new band at 1150 cm⁻¹ correspond to nC-O and Another band located around 1420 cm⁻¹ is assigned $\delta\text{CH}_2 + \delta\text{OCH} + \delta\text{CCH}$. Thermogravimetric analysis (TGA) confirms the binding of glucose there is a loss of weight around 350°C and 410°C corresponds to the decomposition of the glucose. The study of the mossbauer spectrum of FM₈ were carried out the state of oxidation of ferrous ions in montmorillonite structure. This result could confirm that the fixation of glucose was achieved successfully.

Keywords: Montmorillonite KSF, Ferrous ion, Adsorption, Glucose.

INTRODUCTION

There are a wide variety of argillaceous rocks, depending on their structure and mineralogical composition, of which only few are used in medical therapy¹. Each of these clays can, very often, shows some specific therapeutic virtues. Although they present in general a number of similar properties that characterize them.

The montmorillonite studied in the present work are of colloidal clays², which received multiple applications, due to their high exchange capacity and swelling as well as their rheological properties (thixotropy)³. They are therefore used in various fields such as drilling, foundry, ceramics, pharmacy, ... etc.

In the present study we aimed to use a material and a compensating cation in which the retention of glucose is the most important (KSF-Fe²⁺). The kinetics of contact between clay and solutions of glucose was investigated to search for an optimal time giving a better retention. In this article, the results obtained by the combined use of X-ray diffraction, IR, thermal analysis and Mossbauer spectroscopy are described. These analyses were carried out with the objective of determining the state of the iron in montmorillonite structure, and to characterize the interaction between montmorillonite exchanged by ferrous ions and glucose.

EXPERIMENT

Preparation of the adsorbents

All experiments in this work were carried out on a same batch of montmorillonite KSF supplied by Aldrich. It is a commercial montmorillonite, calcium-rich form of gray powder.

The mass ratios of oxides of the elements forming this clay are given in Table 1.

The exchange by Fe²⁺

The cation exchange Fe²⁺ was selected based on its capacity to form a complex with glucose.

In order to investigate the potential fixation of glucose, three materials referenced as FM₃, FM₈ and FM₁₁ were prepared and subjected to a cation exchange with ferrous ions by varying the exchange time as indicated in following chart (Fig.1). The studied materials were then characterized.

Determination of glucose

The determination of glucose was achieved by an enzymatic method reaction, as described previously^{5,6}. The assessments were performed using a spectrophotometer (Shimadzu Corporation, Japan 1200 UV). The peak wavelengths were obtained

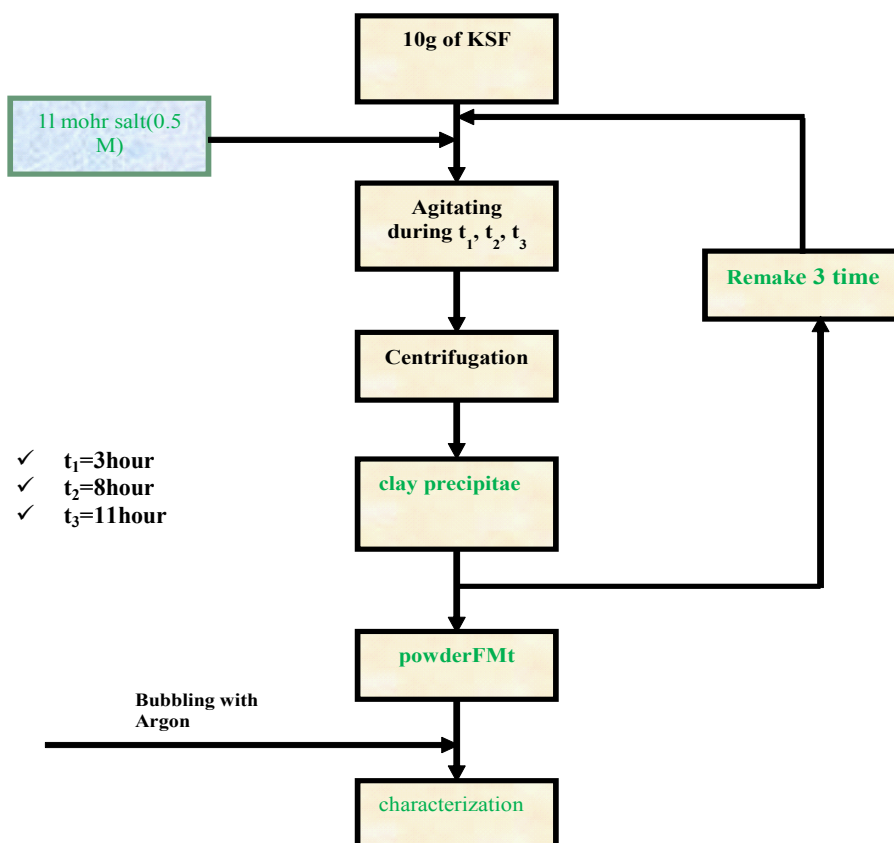
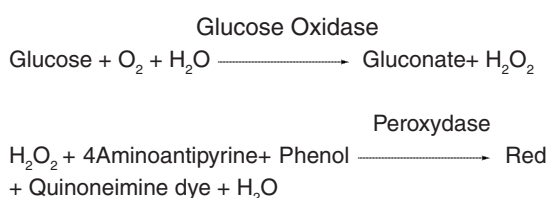


Fig.1: Diagram represents the exchange of KSF by ferrous ions

directly by an automatic scanning between 200 to 800 nm.

Principle

Glucose is oxidised to gluconic acid and hydrogen peroxide in the presence of glucose oxidase. Hydrogen peroxide further reacts with phenol and 4-aminoantipyrine by the catalytic action of peroxidase to form a red coloured quinoneimine dye complex. Intensity of the colour formed is directly proportional to the amount of glucose present in the sample^{7,8,9}.



PROCEDURE

Wavelength: 505 nm
 Temperature: 37°C
 Light path: 1 cm

Addition Sequence **Blank(ml)** **Standard** **Test**

| | | | |
|----------------------|-------|-------|-------|
| Glucose Reagent (L1) | 1(ml) | 1(ml) | 1(ml) |
| Distilled water | 1(μl) | - | - |
| Glucose standard | - | 1(μl) | - |
| Sample | - | - | 1(μl) |

Mix well and incubate at 37°C for 10 mins,

Measure absorbance of the Standard (Abs.S) and Test Sample (Abs.T)

Calculations

$$\text{Total Glucose in mg/dl} = \frac{\text{Abs.T}}{\text{Abs.S}} \times 100$$

Adsorption kinetics

It was necessary to follow the kinetics of the adsorption of glucose to determine the conditions for maximal binding. For this, three suspensions containing 250ml of glucose solution at 2 g/l and 1.5g FM₃, FM₈ and FM₁₁, were kept under stirring followed

Table 1: Chemical composition (in wt %) of montmorillonite KSF⁴

| Oxide | Al ₂ O ₃ | SiO ₂ | Na ₂ O | MgO | P ₂ O ₅ | K ₂ O | CaO | TiO ₂ | MnO | Fe ₂ O ₃ |
|-------|--------------------------------|------------------|-------------------|------|-------------------------------|------------------|------|------------------|------|--------------------------------|
| % | 14.93 | 69.31 | 0.14 | 2.19 | 0.09 | 1.17 | 2.08 | 0.44 | 0.01 | 3.97 |

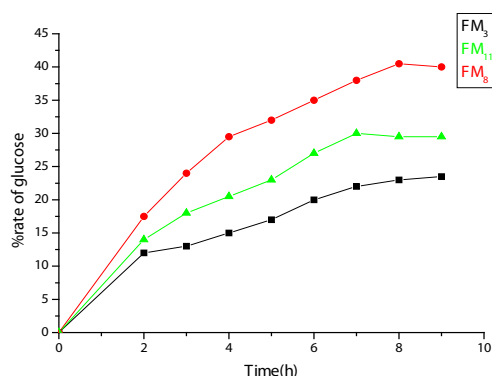


Fig. 2: The % rate of the adsorption of glucose by the studied materials: FM₃, FM₁₁, FM₈

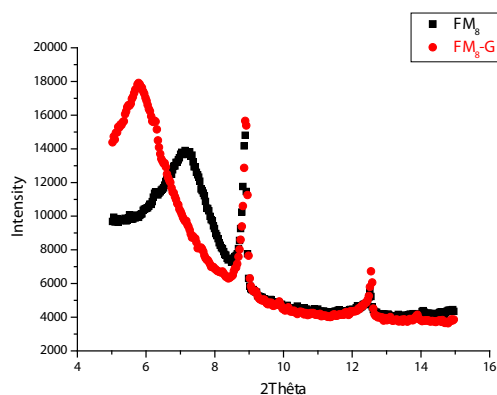


Fig. 3: X-ray powder patterns of the studied samples FM₈ and FM₈-G

by a centrifugation at 4500rpm/min then assayed as described above.

FM₈, and FM₁₁) are 23.5%, 40.5% and 29.5%, respectively.

RESULTS AND DISCUSSIONS

Adsorption kinetics

As shown in the (Fig 2), the maximum adsorption rate for the three materials used (FM₃,

Interestingly, the FM₈ material, prepared using Fe(II) exchanged montmorillonite KSF for an exchange time of 8 hours gives an adsorption rate of 40.5% glucose, which is much higher when

Table 3: Mossbauer parameters at room temperature of FM₈

| FM ₈ | IS, mm/s | QS, mm/s | LW, mm/s | Contribution % |
|-----------------|--------------|--------------|--------------|----------------|
| Comp1 | 1.09 ± 0.02 | 2.00 ± 0.03 | 0.32 ± 0.03 | 13 ± 5 |
| Comp2 | 1.06 ± -1.00 | 2.82 ± -1.00 | 0.23 ± -1.00 | 2 ± -1 |
| Comp3 | 0.36 ± 0.00 | 1.21 ± 0.01 | 0.30 ± 0.02 | 38 ± 5 |
| Comp4 | 0.36 ± 0.01 | 0.68 ± 0.07 | 0.56 ± 0.06 | 34 ± 7 |
| Comp5 | 0.44 ± 0.01 | 0.22 ± 0.03 | 0.31 ± 0.08 | 12 ± 4 |

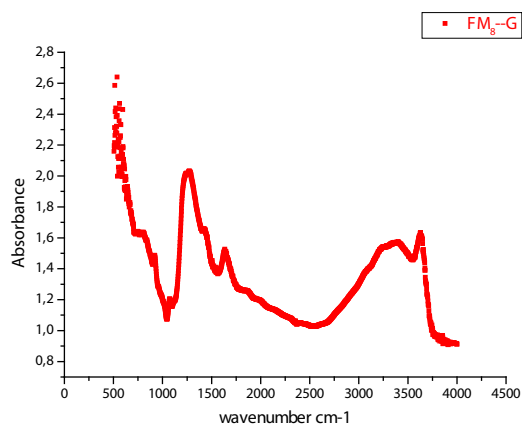


Fig. 4: Infrared spectrum of FM₈-G

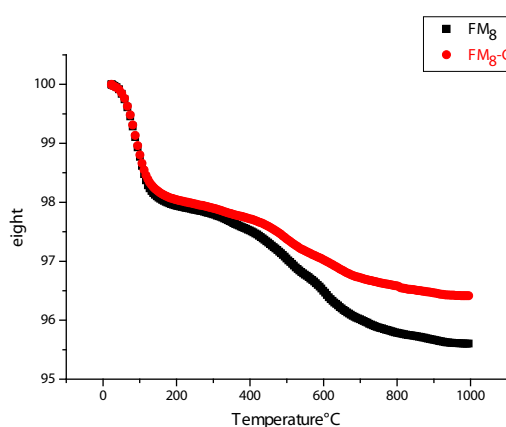


Fig. 5. Thermal gravimetric analysis of studied samples FM₈ and FM₈-G

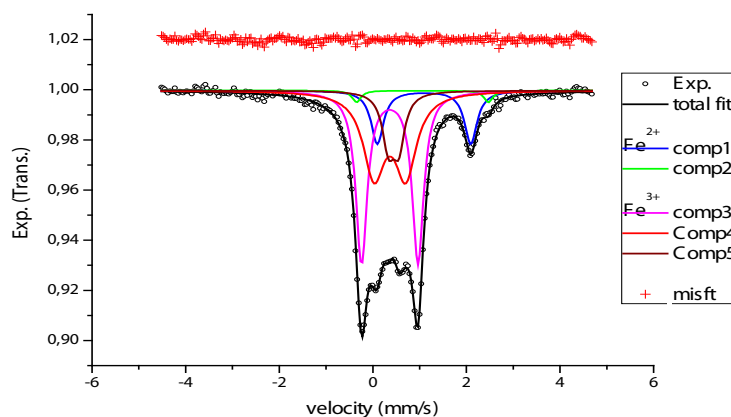


Fig. 6: Room temperature Mossbauer spectrum of FM8 sample

compared to those obtained with the other two materials (FM_3 and FM_{11}).

Characterization by XRD

Powder pattern was recorded on a PHILIPS X'Pert MPD λ - λ diffractometer equipped with the X'Celerator detector with Cu K α radiation ($\lambda = 1.5418 \text{ \AA}$) and nickel filter. The different results regarding the XRD characterization of the studied samples are summarized in Fig. 3. Examination of this recorded diffractogram and peak list show that the basic structure of the aluminosilicates is maintained and the values of angle are bands q2 Characteristics of montmorillonite. It seems clearly that the distance d_{001} of exchanged montmorillonite by iron ions affected a slight change in the inter-fol layer distance after the fixation of glucose.

Characterization by IR

The use of infrared spectroscopy allows determining information about the different absorption bands existing in the material FM_8 -G. The IR spectrum examination (Fig. 4) shows the following bands: An intense strip at 3450 cm^{-1} and a strip at about 1630 cm^{-1} ; Are relative to the modes of vibration of the water molecules adsorbed in the inter-sheet space^{10,11,12}, the bands located around 1050 cm^{-1} and 910 cm^{-1} and 840 cm^{-1} are assigned respectively to the "Elongation of the Si-O bond; The deformation of the Al-Al-OH bond and the deformation of the Al-Mg-OH bond, the band located around 780 cm^{-1} is due to the elongation vibration of the bond Si-O bond^{13,14}, we noticed an appearance of new band at 1150 cm^{-1} correspond to C-O and Another band located around 1420 cm^{-1} is assigned $\delta CH_2 + \delta OCH + \delta CCH$.

This result could confirm that the fixation of glucose was achieved successfully.

Thermal gravimetric analysis

The process of weight loss can be divided into two stages the first weight loss observed around 100°C is attributed to the desorption of water (with may be physical or chemical adsorption on

the interparticle surface of two samples (FM_8 and FM_8 -G), the second step from 560 to 600°C with the main weight loss can correspond to the weight loss caused by breaking the Calcite¹⁵. In the sample FM_8 -G there is a loss of weight around 350°C and 410°C corresponds to the decomposition of the glucose.

Mossbauer analysis

A typical Mossbauer spectrum of FM_8 is shown in (Fig. 6), whereas the computer-fitted parameters for the sample studied are given in Table 3.

Iron is present in 5 forms, 2 Fe^{2+} kinds (15%) and 3 types of Fe^{3+} (85%)

CONCLUSION

- This study describes the preparation of a new material based on clay: montmorillonite Fe^{2+} shows a high percentage of glucose capture.
- The results showed an important adsorption of glucose (around 40,5%) with compensating cation ($Fe(II)$ - KSF) at 8 hours where the exchange of montmorillonite KSF with ferrous ions. This sample is identified as FM_8 .
- The use of infrared spectroscopy allows determining information about the different absorption bands existing, appearance of new band at 1150 cm^{-1} correspond to $\nu C-O$ and Another band located around 1420 cm^{-1} is assigned $\delta CH_2 + \delta OCH + \delta CCH$.
- The thermogravimetric analysis makes it possible to follow the loss of mass of the sample as a function of the temperature, In the sample FM_8 -G there is a loss of weight around 350°C , 410°C corresponds to the decomposition of the glucose
- Mossbauer spectroscopy was carried out to determine the state of oxidation of ferrous ions in montmorillonite structure.
- This result could confirm that the fixation of glucose was achieved successfully.

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