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Research on preparation and filtering effects of modified talc filter aid

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ABSTRACT

To reduce the shortcomings of existing filtering assistance, the talc was modified by a two-step method. In the first step, the talcum powder was heated by microwave in the acid solution. In the second step, the talcum powder was stirred with Hexadecyl Trimethyl Ammonium Bromide (HTAB) solution for optimum time under an optimum temperature. The optimum temperature, time and amount of HTAB were investigated by the batch membrane filtration experiments of kaolin suspension. The filtering effects of the new talc filter aid were evaluated with the filtration rate and moisture content in filter cake. The results showed that the optimum conditions were: the amount of 0.05 mmol/L HTAB was 100 ml, the modified temperature was 80°C and the modified time was 60 min. When the filtrate was accumulated to 25 ml, the filtration time was 13.21 s shorter than others. The membrane flux of microfiltration was the largest (0.024 L/m^2·s) when new talc filter aid was added into the kaolin suspension. The talc powder was characterized by Fourier Transform Infrared Spectroscopy and X-ray diffraction, which made the experimental results more obvious. Characterization results indicated that the hydrophobicity of the talc filter aid was excellent because microwave-assisted acidification talcum powder could absorb the cationic surfactants by electrostatic interactions. Finally, by comparing talc filtration assist with diatomite filtration assist, it is concluded that the filtration performance of talc filtration assist kaolin suspension is superior to other materials.

Keywords: Talcum powder; Modification; Cationic surfactants; Filter aid; Microwave.

1. INTRODUCTION

With the development of the industry, more and more things need filters, including food processing, drugs, water treatment, chemical products, etc. In the filtering process, it is necessary to use a large number of filtering aids to achieve it. Excellent filter aids should not contain soluble impurities. In addition, excellent filter aid can accelerate the filtration rate and adsorption of fine solid particles. In recent years, filtering aids have focused on diatomite, perlite, cellulose, asbestos, and active carbon. Still, these filter aids have many obvious drawbacks [1]. For one thing, due to the high price of diatomite ore and complex production technology, the production process will pollute the environment. Although the retained reserves of China rank second in the world, diatomite research started late, the gap between functional materials compared with foreign countries, and the production level is low and single [2–3]. For another thing, diatomite filters often fail to achieve the desired effect due to the high density of diatomite adding more will increase the cost [4–5]. Although bead filter density is low, low porous density, low cost but poor chemical stability, filter liquid clarity is not perfect, complex production process and harsh conditions [6]. Asbestos is a natural fiber filter aid, but filtering is not great when used alone. In addition, constant exposure to asbestos can harm humans [7]. Cellulose filtration aids are an organic material that does not damage equipment and human health. But the processing process is complex, the physical treatment performance is unstable, the chemical treatment affects the environment, and the biological treatment efficiency is low [8]. In general, the main problems of filter aids include the environmental impact of raw material processing, high production cost, poor chemical stability, and high price. Therefore, it is particularly important to develop a new filter agent with good chemical stability, less pollution, low cost and good filtration effect.

Talc is monoclinic with a layered structure. Its crystal structure is a tetrahedron composed of two layers of silicon atoms and oxygen atoms sandwiched by an octahedron composed of magnesium atoms, oxygen atoms and hydroxyl atoms, with a thickness of 10Å (Figure 1) [9]. Its cationic structure which consists of [$MgO_4(OH)_2$] is

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a trioctahedron. And the oxygen atoms shared by the Si-O tetrahedron are held together in a continuous network structure. All the activated oxygen atoms are toward one side. The double Si-O tetrahedrons are linked together by an octahedron of magnesium oxide, as shown in Figure 2 [10–11]. Moreover, the charge getting to balance in the talc unit layer and the interlayer are bonded together by weak Van der Waals' force, easy to peel under the action of external forces. The crystal structure of talc shows that it has good chemical stability. Meanwhile, talc has a unique pore structure, excellent lubricity and other properties, such as rich resources and low costs [12–13]. Talc belongs to the natural magnesium silicate mineral, with a unique layered structure, large specific surface area, extremely strong adsorption capacity, good electrical insulation, and stable chemical properties. Talc has obvious advantages in price, processing cost and yield although its diameter thickness ratio is smaller than mica. The interlayer connection of talc molecules (van der Waals force) is very weak, so that its hardness is low, the process of equipment wear is less, but also can give the best balance between the stiffness and impact strength of the filling system. So talc has been widely applied in papermaking, coating, cosmetics and other industries [14–15].

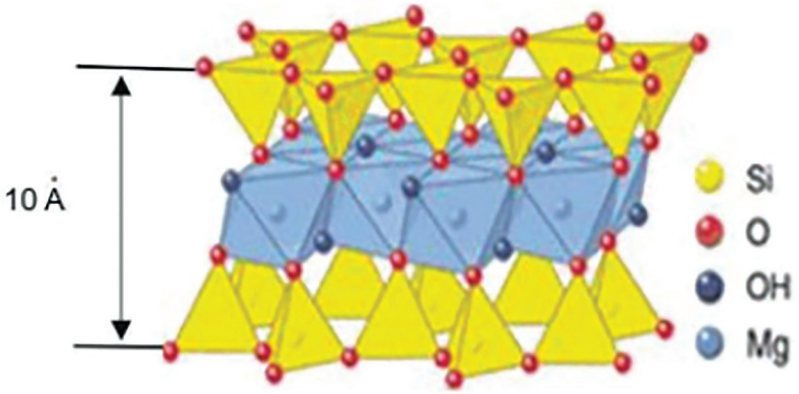


Figure 1: Crystal structure of talc.

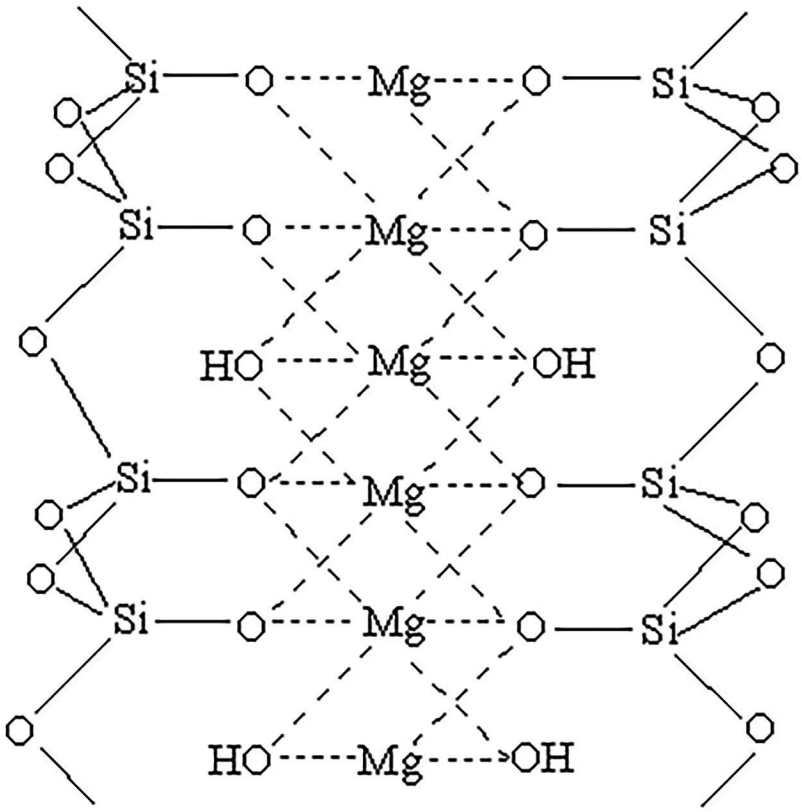


Figure 2: Schematic diagram of the crystal structure of talc.

Studies on talc powder modified by acetic acid show that the modified talc powder has greatly improved impact properties and tensile properties, reduced dimensional change rate, improved stability, and strong interface bonding [16]. Another study indicated that talc was modified by hydrochloric, formic and acetic acid, respectively, were purer and the associated minerals were removed completely. The hydrochloric modified talc was hydrophilic and the organic acid modified talc was hydrophobic [17]. Some studies have also shown that the properties of talc modified by acidification are better and the purity of talc modified by surface is greatly increased [18].

Microwave heating is a heating method using magnetic field interaction between polar molecules and non-polar molecules. In this process, molecules have to rearrange with the changing direction of high-frequency electric field, and molecules need to overcome the interference and obstruction of original thermal motion and molecular interaction, thus producing an effect similar to friction. The whole process gradually converts electromagnetic energy into new heat energy, but does not damage the molecular structure itself [19]. Furthermore, modification by microwave heating is more rapid and more uniform, significantly reducing heating time and energy consumption [20]. And Microwave-assisted vitriol modification could remove impurities and moisture of diatomite [21]. Meanwhile, silicate minerals have been modified with cationic surfactants by some researchers in order to improve the performance of adsorption and hydrophobic in recent years. For example, Yang Futing et al used several cationic surfactants (Cetyltrimethylammonium Chloride, Dodecyltrimethylammonium Bromide, Octadecy trimethyl ammonium bromide) to modify talcum powder. The results showed that the pollutants with a negative charge in deinking wastewater were neutralized by talcum powder after organic modification. So the COD (Chemical Oxygen Demand) removal efficiency was increased greatly [22]. The study by Lu Yiping et al revealed that the dispersion stability of cationic surfactant triethanolamine on ultrafine talc was poor. The electrostatic repulsion effect decreased and then the particles of talc gathered easily [23]. Based on modification methods of talcum powder and the characteristics of filter aid, the talc powder was modified by a two-step method. First, to improve the activity of certain groups and remove impurities and moisture, the talc powder will be treated with microwave and acetic acid. Second, the above talcum powder was modified by HTAB. Figure 3 showed the structure of HTAB. HTAB is composed of a hydrophobic hydrocarbon chain and a hydrophilic polar group. It is a cationic surfactant with high hydrophobic ability. And the hydrophobicity of talcum powder was improved in this step, meanwhile, the optimum conditions of modified time, modified temperature and the amount of HTAB were also determined in this step. Then the talc filter aid prepared by the two-step method is applied to the filtration experiments of kaolin suspension. The performance of the talc filter aid was valued by filtration rate and moisture content in filter cake. The original and the talc filter aid were characterized by the Fourier Transform Infrared Spectroscopy, X-ray diffraction [15].

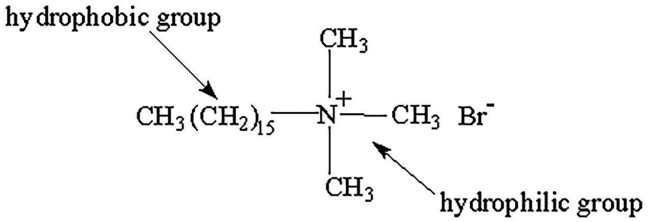


Figure 3: The structure of HTAB.

2. EXPERIMENTAL

2.1. Materials

The Talcum powder (1250 mesh) samples from Liaoning Haicheng Talcum powder Company (Haicheng City, China), acetic acid (Analytical Pure) 99.5% purity, hexadecyl trimethyl ammonium bromide (HTAB)(chemical formula: $C_{19}H_{42}BrN$, molecular weight: 364.45), kaolin (chemically pure, chemical formula: $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$, molecular weight: 258.16), diatomite (chemically pure), Silver nitrate(chemical formula $AgNO_3$, molecular weight: 169.87), hybrid fiber microfiltration membrane (Φ80*0.45 μm) from Sinopharm Chemical Reagent CO, and LTD of Shanghai were used in this experiment.

2.2. Microwave assisted acetic acid modification of talcum powder

The talc powder samples should be treated as follows before experimental use: Step 1: Clean the sample,1250 mesh talcum powder was mixed with 300 ml distilled water, then the mixtures were stirred continuously for 1 h.

Step 2: After stirring, all mixtures need to stand for 1 h in order to remove the small impurity by pipette. This process was repeated many times until there were not suspension matters which could be seen by eyes in the solution. Step 3: The cleaned talcum powder was then dried at 105°C. For acidification, 10 g of washed talc powder samples were placed into 100 ml of the acetic acid solution for 40°C for 9 h. Step 4: These suspensions were treated by the MAS-I microwave synthesis system which was from Shanghai Sineo Microwave Chemical Technology Co, Ltd. for 5 min on 600 W working power afterward. Step 5: Then samples were dried at 120°C and marked with M-A-T.

2.3. HTAB modified M-A-T

HTAB surface modification:5 mmol/L HTAB solution and 5 g M-A-T samples were stirred at different time (40, 60, 80, 100 min) at different temperatures (60, 80, 90°C). Filtrate the solution obtained at different time and temperature to obtain white precipitation. The white precipitate is then washed with distilled water until there are no Br- ions in the precipitate. Finally, the obtained samples were dried at 90 ± 5°C and labeled H-M-A-T.

2.4. Characterization

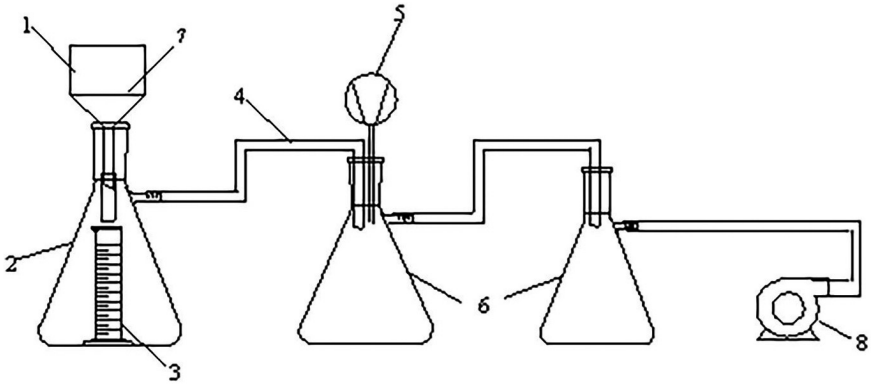
The chemical bonds before and after modification talcum powder were measured by pectrum Two Fourier Transform Infrared Spectroscopy (FTIR) which is from Perkin Elmer Company (American). Spectra were obtained in the range 400 to 4000 cm^{-1}. The phase constitution of before and after modification talcum powder was identified by X-ray Diffraction (XRD) which in a D/max RB diffract meter. Getting diffraction patterns in a D/max RB diffractometer (Japan) at 40 Kv and 30 mA. The values of 2θ ranged from 5° to 70°.

2.5. Batch filtration experiments

Batch filtration experiment was conducted with the equipment in Figure 4. Soak the hybrid fiber microfiltration membrane in 70°C degrees of distilled water for 4 h, and then soak it in another beaker for 12 h. The filter unit shown in Figure 4. The addition of 0.0500 g of H-M-A-T to a 40 ml of kaolin suspension gave the mixture an 8% mass fraction, stirring for 3 min. Adjust the vacuum pump pressure to constant pressure of 0.09 mpa, then transfer the mixture to Buchner funnel, start to calculate the filtration time, let the mixture filter through the microfiltration membrane, accumulate to 5 ml each time. After the process of filtration, the filter cake were placed in the dry watch glass, weighed, and dried to constant weight. The water content and membrane flux were calculated and calculated as follows.

$$J = \frac{V}{T \bullet S}$$

Where J is membrane flux ($L/m^2 \cdot s$), V is volume of filtrate out of funnel (L), T is filtration time (s), S is active area of membrane (m^2).



1. Buchner funnel, 2. 2500 ml filter flask, 3. 25 ml measuring culinder, 4. Rubber hose,

5. Pressure gage, 6. Buffer bottle, 7. Microfiltration membrane, 8. Vacuum pump

Figure 4: Filter unit of kaolin suspension.

3. RESULTS AND DISCUSSION

3.1. Effect of modified temperature on talc filter aid in filtering process

Figure 5 and Table 1 illustrates that the filtration rate of kaolin suspension reached the highest at 80℃. At the same time, the moisture content in the filter cake was at the lowest. At 80℃, when the filtrate was accumulated to 25 ml, the time was 79.32 s, which was 48.73 s faster than that without the filter aid (blank test). The above results probably were due to the low reaction rate at 60℃. With the temperature rising, the filtration speed gradually increases, on one hand, abundant energy was provided, and then the chemical adsorption between M-A-T and cationic surfactant happened. For another, as the reaction temperature increases. The accelerated intermolecular movement increases the contact area, and then increases the reaction probability. When the temperature rises above 90℃, the suspension faces evaporation, which will greatly affect the experimental results. Therefore, the optimal modification temperature was determined to be 80℃.

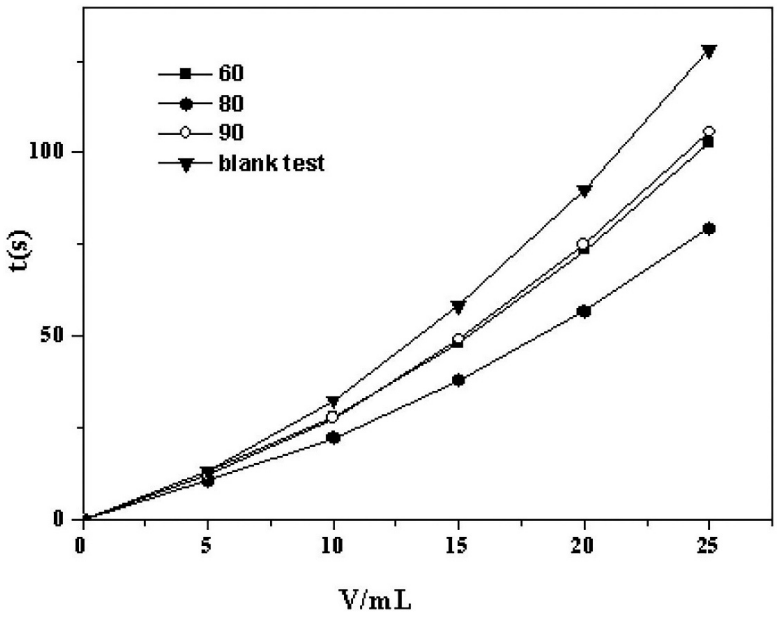


Figure 5: Effect of modified temperature on talc filter aid in filtering process.

Table 1: Effect of modified temperature on the moisture content in filter cake.

Modified temperature(℃)	60	80	90	Blank test
moisture content(%)	36.49	34.66	36.58	38.02

3.2. Effect of modified time on talc filter aid in filtering process

From Figure 6 and Table 2, the results showed that the filtration rate of kaolin suspension gradually increased with the modification time, and reached the highest value at 60 min, while the moisture content of filter cake was the lowest. After more than 60 min, the filtration rate gradually decreases, but the filtration rate with talc filtration assistance is faster than that without talc filtration assistance. It can be understood that the reaction of HTAB and M-A-T is not thorough enough in a short time, and the adsorption of M-A-T over time reaches equilibrium, resulting in the disappearance of the active site, and the final adsorption capacity no longer changes. Furthermore, an emulsion also appeared in these reactants easily when the stirred time was too long, then the hydrophobic ability of talc filter aid would decrease. This will reduce the filtration rate of kaolin suspension. So the optimum modified time was 60 min.

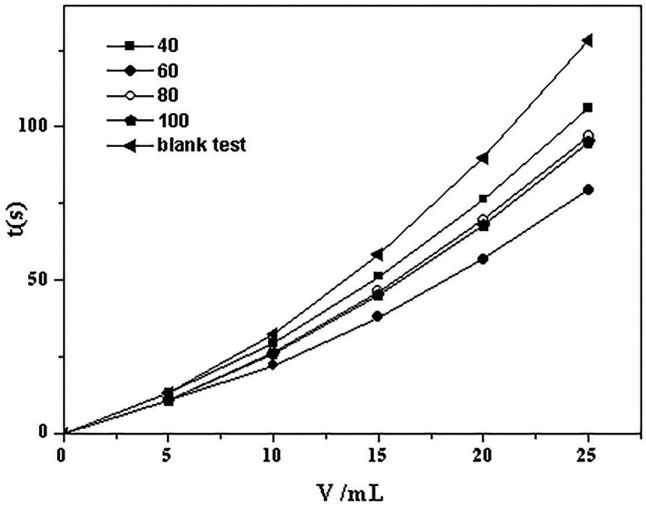


Figure 6: Effect of modified time on talc filter aid in filtering process.

Table 2: Effect of modified time on the moisture content in filter cake.

Modified time(min)	40	60	80	100	Blank test
moisture content(%)	36.52	34.66	35.94	35.89	38.02

3.3. Effect of the amount of HTAB on talc filter aid in filtering process

The results are given in Figure 7 and Table 3. It was noted that when 100 ml HTAB was added, the filtration rate of kaolin suspension reached the highest, and the moisture content in filter cake was the lowest. But the decreasing filtration rate occurred when HTAB was continued to add. The filtration rate curve gradually stabilized when the HTAB content was greater than 200 ml. The reason was that the M-A-T adsorption on cationic surfactants was already saturated when the amount of HTAB was 200 ml or more. Meanwhile, a lot of froth was formed within the stirring time. And there were parts of water molecules went into the spaces in the adsorption process, so the hydrophobic ability of talc filter aid decreased. This has no positive effect on examining the filtering performance. Hence, the best amount of DTAB was 100 ml.

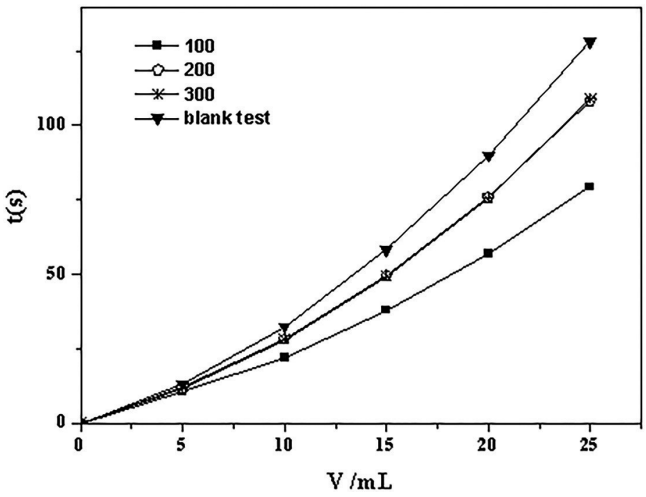


Figure 7: Effect of the amount of HTAB on talc filter aid in filtering process.

Table 3: Effect of the amount of HTAB on the moisture content in filter cake.

The amount of HTAB(ml)	100	200	300	Blank test
moisture content(%)	34.66	37.42	37.39	38.02

3.4. Characterization of talcum powder before and after modified and its modified mechanism

FTIR spectra

Infrared spectroscopy is one of the most effective methods used for compound identification [24]. For oxides, the surface structure is closely related to many applications (such as catalysis, biomedical, etc.). And these surface hydroxyl groups using XRD is certainly uncertain, this time using infrared characterization has advantages, especially in situ infrared, can study the change of surface hydroxyl groups at different temperatures, and then related to its performance [25]. The FTIR spectra from original talcum powder, M-A-T and H-M-A-T are presented in Figure 8. An analysis of the original talcum powder was done and presented in Table 4 [19–26]. The peak area of Mg-O bonds (463 cm^{-1}) and Si-O-Mg bonds (539 cm^{-1}) were both decreased. It showed that in layer structure of M-A-T, the bonding interaction was influenced. And because part of Mg-O bonds was broken with microwave radiant energy, and this made the binding force weak in the interlayer, then the $Si\text{-}O^-$ and Mg^+ exposed. The active groups such as Si-O- and OH^- from the edge surface laid the foundation for the reaction between HTAB and M-A-T. Meanwhile, the peak area of absorbing water from M-A-T was larger. The most likely reason was that more –OH was exposed after the break of Mg-O bonds and Si-O-Mg bonds, and then they formed hydrogen bonds with water molecules.

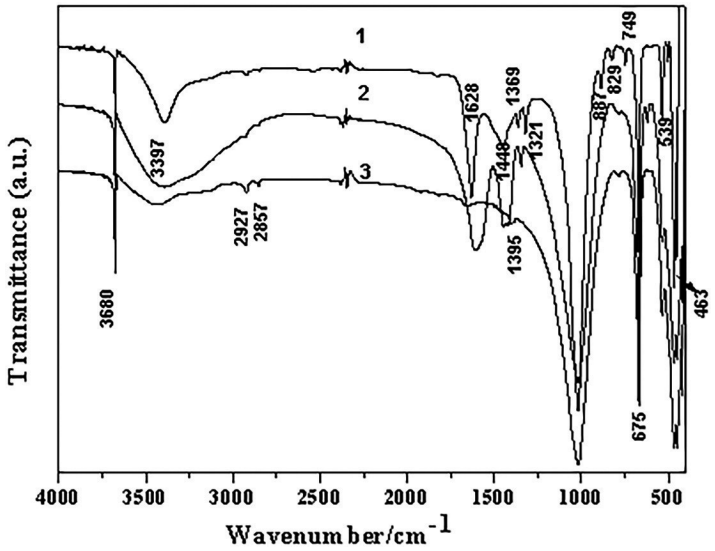


Figure 8: FTIR spectra of talcum powder: 1 unmodified talcum powder ,2 M-A-T and 3 H-M-A-T.

Table 4: Peak position in FTIR spectra of original talcum powder.

WAVE NUMBER (cm^{-1})	SPECTRAL ANALYSIS
3680	Mg_3-OH
3997 1628	-OH, stretching -OH, bending
1448,829	Dolomite, Magnesite
1369,1321	CO_3^{2-} , stretching
1016	Si-O-Si, stretching
887,749	Magnesite
675	-OH, bending
539	Si -O –Mg, stretching
463	Mg-O

Moreover, the absorption peak of carbonate minerals decreased, or even disappeared. It also indicated that the M-A-T samples were more pure, which probably due to acetate removal of associated minerals from the talc pore, making the talc purer and smaller particle size [17]. But there were some other impurities in M-A-T.

From Figure 7, the bands at 2927 cm^{-1}, 2857 cm^{-1} appeared, which could be stretching vibration or antisymmetric stretching vibration of $–CH_2–$, an antisymmetric vibration of $–CH_3–$ was appeared at 1395 cm^{-1}. Electron donor O^- , which exposed on the edge surface of M-A-T, were reacted with electron deficiency $H_3C(H_2C)_{15}N^+(CH_3)_3$ by electrostatic interactions, then $H_3C(H_2C)_{15}N^+(CH_3)_3$ were attached to the edge surface of talcum powder. But the effects might be not strong because of the influence of $–CH_3–$ on $–\overset{|}{N^+}–$. Also, the bands (3997 cm^{-1},1628 cm^{-1}) of absorbed water in the interlayer were decreased obviously, it showed the hydrophobic property of H-M-A-T was improved greatly. The reason most likely was the groups ($OH^–$ and $Si\text{-}O^–$) on the edge surface of M-A-T attached to a number of the hydrophobic long carbon chains of HTAB. It is also probably because the hydrophobic groups Si-O-Si on face surface of H-M-A-T and hydrophobic groups of HTAB are combined by hydrophobic interaction. As shown in Figure 9, there are stable chemical bond in the interior of H-M-A-T. Around the surface of H-M-A-T, many hydrophobic carbon chains of HTAB gather irregularly, increasing the hydrophobic area, so that water molecules stay away from H-M-A-T [27]. From what has been discussed above, it would be reasonable to show that the hydrophobic area increased in H-M-A-T particles. This contributed to decreasing the moisture content in filter cake and getting through the fluid channels of filtrate [28–29].

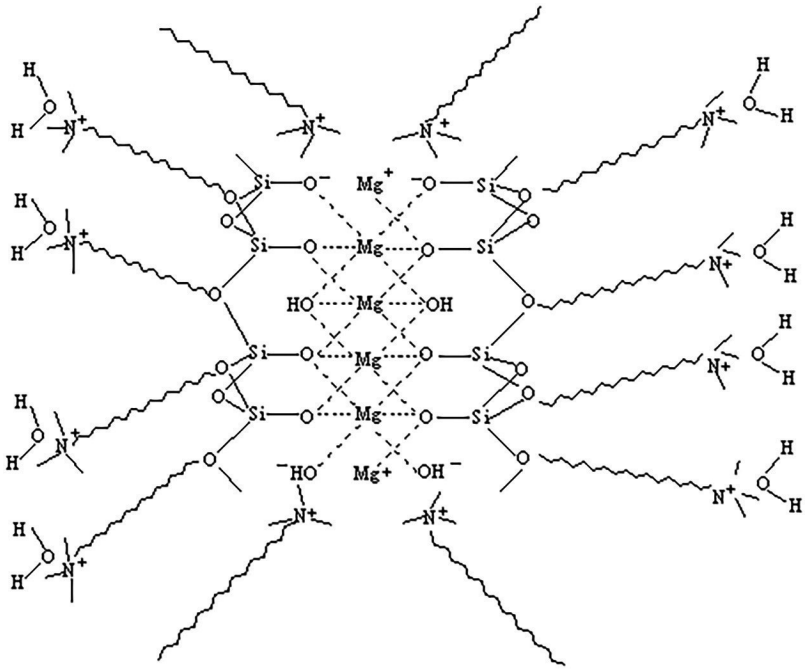


Figure 9: The electrostatic interactions of M-A-T and HTAB and hydrophobic interaction of groups of face surface.

Moreover, the impurities that remained in M-A-T were removed completely by HTAB. To some extent, this illustrated alkaline ammonium ion of HTAB removed the carbonate. The filtration rate became faster in the filtering process because the intragranular pores and the spaces of H-M-A-T particles increased, and the channels of H-M-A-T were broadened. The filtrate was more clarity by the filtration effect and adsorption effect of the talc filter aid.

3.5. X-Ray diffraction

The combination of infrared spectrum and XRD is more helpful to the qualitative analysis of samples, to determine the material composition, and the experiment is more scientific [30]. The results of characterization by XRD of original talcum powder, M-A-T and H-M-A-T were presented in Figure 10. The diffraction angle which indicated the typical diffraction peak of original talcum powder was 9.451°, 18.988°and 28.586°. The interplanar spacing d (Å) corresponding to d(002), d(004), d(006) was 9.35, 4.67 and 3.12, respectively. The intensity of the typical diffraction peak of M-A-T was significantly reduced compared to the unmodified talc powder, and, in contrast, the interlayer and structural water also disappeared. There is a slight damage to the layer structure under microwave radiation. Meanwhile, the M-A-T surface has exposed more pores. The pores were preserved in the step of preparation of H-M-A-T, again, talcum powder itself was porous, so the talc filter aid could retain kaolin particles effectively and reduce pollution of millipore filter in the filtering process.

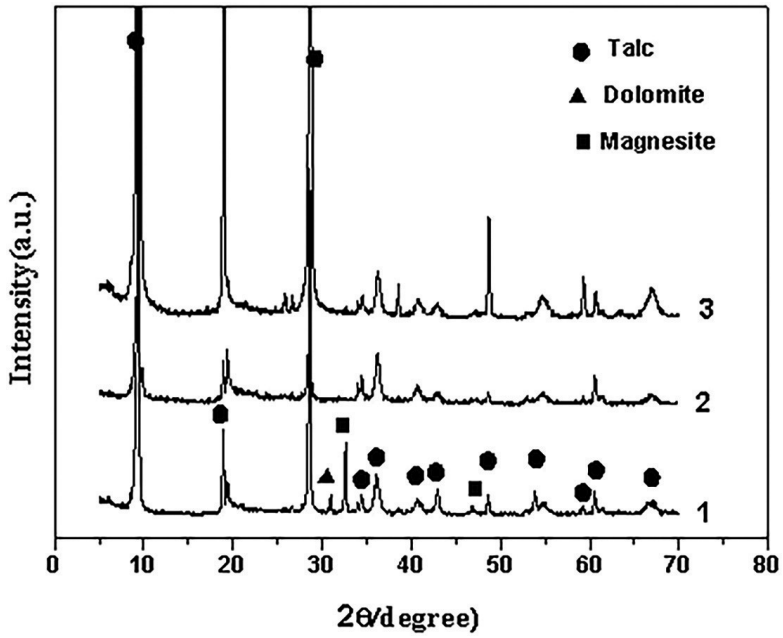


Figure 10: XRD spectrum of talcum powder: 1 original talcum powder (Max. intensity=16593 counts), 2 M-A-T (Max. intensity=7580 counts) and 3 H-M-A-T (Max. intensity=29203 counts).

The interplanar spacing d(002) and d(110) were reduced slightly, it showed that $-N^{+}-$ did not enter into the interlayer of talcum powder. The results might because the water in the interlayer was taken off when the modifier contacted with an interlayer of talcum powder in the heating process. This could enhance the hydrophobic property of the talc filter aid. Then the filtering effect was excellent, this was consistent with the analysis results of FTIR.

The intermolecular forces of talcum powder were probably dispersed when the cation of HTAB reacted with $Si\text{-}O^{-}$ of M-A-T by electrostatic interactions. It resulted in sliding easily in the layers of H-M-A-T. And HTAB itself was lubrication, hence, the lubricity of talc filter aid probably increased. Moreover, according to the formula (1), the membrane flux of kaolin suspension with talc filter, diatomite filter aid and without talc filter aid (blank test) were, respectively, 8.377×10^{-3}, 0.024, 0.010 $L/m^2\cdot s$. It can be seen that the membrane flux was the largest with talc filter aid. Therefore, the filtration rate of kaolin suspension with talc filter aid was faster.

As can be seen from the Figure 8, the diffraction peaks of dolomite (2θ = 30.962°) and magnesite (2θ = 32.629°) were both disappeared completely in M-A-T. The diffraction peaks of magnesite (2θ = 46.815°) of original talcum powder were disappeared completely after HTAB modification. Meanwhile, the diffraction peaks of impurities reduced significantly or even disappeared. The intensity of characteristic diffraction peaks of talcum powder in H-M-A-T was increased. These performances corresponded to the results of the FTIR analysis. Thus it can be seen that the talc filter aid was more pure.

3.6. A comparison of talc filter aid with diatomite filter aid

Diatomite filter aid has more accurate filter effect than perlite, healthier than asbestos, lower cost than cellulose, and rich reserves [2–8]. Therefore, diatomite is the most popular filter aid in the world today, which has a similar structure to the HTAB-talc filter aid. By comparing the high quality talc and diatomite filters to the kaolin suspension. A comparison of Figure 11 shows that the rate of talc filtration assistance is almost as fast as that of diatomite filtration assistance when the filtrate accumulated to 5 ml. However, as the filtrate increased, the filtration time gradually shortened with talc filter aid. When the filtrate was accumulated to 25 ml, the filtration time was faster 13.21 s with talc filter aid than that with diatomite filter aid and 48.73 s than that without any filter aid. And thus, the performances of filtration of kaolin suspension with talc filter aid were better than others'. Meanwhile, its filter cake was dry. Besides, the talc filter aid which was prepared with the two step methods was more pure and free of other impurities. The hydrophobicity and lubricity of H-M-A-T were both improved. Talc filter aid was an ideal filter aid.

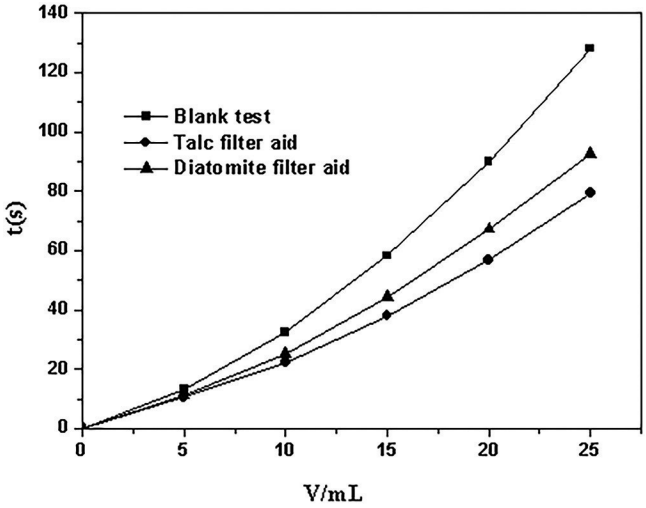


Figure 11: Effect on filtration rate of talc filter aid and diatomite filter aid.

4. CONCLUSION

(1) By studying the changes of the new talc filter aid (Microwave-assisted acidification followed by HTAB modification was performed) on kaolin suspension filtration. Talc powder filter aid has a good filtration effect compared with diatomite filter aid. When the filtrate accumulates to 25 ml, talc filtration time is 13.21 s faster than diatomite and 48.73 s faster than helpless filtration time. Meanwhile, the membrane flux of microfiltration was the largest (0.024 $L/m^2 \cdot s$) when added talc filter aid into the kaolin suspension. Meanwhile, the optimum conditions that HTAB reacted with M-A-T were 80°C, 60 min and 100 ml HTAB.

(2) The results of characterization by FTIR and XRD were showed that the Si-O-Mg bonds from the edge surface of talcum powder which modified by microwave and acid were broken into $Si\text{-}O^-$ leading to more adsorption sites with higher activity. The dolomite impurities were removed completely. Chemical adsorption was formed between the groups $Si\text{-}O^-$ and OH^- on the edge of M-A-T and $H_3C(H_2C)_{15}N^+(CH_3)_3$ of HTAB by electrostatic interactions. The talc filter aid had higher hydrophobicity. And the filter cake had lower moisture content and was loose. The new talc filter aid could accelerate the filtration rate and provide a lot of fluid channels to filter kaolin suspension.

(3) Appropriate modified talc powder is an excellent water treatment agent with good filtration effect. Talc powder reserves, cheap preparation, simple operation, non-toxic and stable chemical properties, can obviously accelerate the filtration rate, and no shortcomings of filtration auxiliary agent found, can be widely used in the filtration auxiliary industry in the future.

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