Effect and mechanism of siderite on reverse flotation of hematite

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Abstract: The effects of siderite on reverse flotation of hematite were investigated using micro flotation, adsorption tests, and Fourier transform infrared spectroscopy. The flotation results show that interactions between siderite and quartz are the main reasons that siderite significantly influences the floatability. The interactions are attributed to dissolved siderite species and fine siderite particles. The interaction due to the dissolved species is, however, dominant. Derjaguin-Landau-Verwey-Overbeek (DLVO) theoretical calculations reveal that adhesion on quartz increases when the siderite particle size decreases and that fine particles partly influence quartz floatability. Chemical solution calculations indicate that the dissolved species of siderite might convert the surface of active quartz to CaCO₃ precipitates that can be depressed by starch. The theoretical calculations are in good agreement with the results of adsorption tests and FTIR spectroscopy and explain the reasons why siderite significantly influences reverse flotation of hematite.

Keywords: hematite; siderite; reverse flotation; solution chemistry

1. Introduction

Flotation based on the surface-chemistry theory serves as the primary method for recovering hematite. Reverse flotation is the main process due to its higher separation efficiency and concentrate quality compared with the direct flotation. Oleic acid and amine are commonly used as collectors, and starch and its derivatives are effective depressants in hematite reverse flotation [1-4]. Hematite associated with siderite or other carbonate minerals is generally referred to as refractory hematite containing carbonates and is commonly found in Donganshan iron ore deposits. Production practice shows that the existence of siderite significantly influences the reverse flotation of hematite and decreases the grade and recovery of concentrate $[5-6]$. A novel two-step flotation process was developed for separation of refractory hematite containing carbonates, which may reduce the negative influence of siderite by removing it at a neutral pH value before the reverse flotation $[7-8]$. It is generally believed that the hardness of siderite is low, a fact leading it to become slime during ore dressing. Because slime has good adhesion on other minerals, it results in the similar floatability for quartz, hematite minerals, and siderite, which brings out the difficulty of separation $[9-10]$.

However, the content of siderite in hematite containing carbonates is relatively low, which limits the effects of adhesion on the other minerals. In addition, coarse siderite can also influence flotation. This contradiction indicates that there is a problem in the adhesion of siderite slime to explain why siderite significantly affects reverse flotation of hematite. As a salt-type mineral, siderite has a relatively high solubility. The dissolved mineral species can undergo some reactions such as hydrolysis, adsorption, and surface and bulk precipitations, which might inhibit selective interactions between reagents and minerals $[11-14]$. However, the effects of dissolved species, especially, its potential effect on reverse flotation of hematite, are neglected in previous works. Therefore, it is necessary to investigate the effects of siderite on the floatability.

In this work, the effects of siderite on reverse flotation of hematite were investigated by micro flotation, adsorption tests, and Fourier transform infrared spectroscopy (FTIR). The mechanism of siderite effects was discussed according to the analysis of Derjaguin-Landau-Verwey-Overbeek

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(DLVO) theoretical calculations and solution chemistry. The results are beneficial for separation of refractory hematite containing carbonates.

2. Experimental

2.1. Materials

Minerals used in this work contain hematite, siderite, and quartz and were obtained from Anshan in Liaoning Province, China. Hand-selected hematite and quartz were crushed and ground to $-106 + 45$ µm in a laboratory ceramic ball mill. Siderite was treated with a similar procedure and sieved to -18 µm and $-106 + 45$ µm, respectively. The purity of the minerals was examined by X-ray diffraction (XRD) and chemical composition analysis. The results show that the mineral purity (hematite and quartz $> 95\%$, siderite > 90%) meets experimental requirements. All chemical reagents were of analytical grade. Distilled water was produced by an automatic adsorption-type ultra-pure water system.

2.2. Micro flotation tests

Single mineral flotation was performed on a laboratory XFD series flotation machine at a speed of 1750 r/min. A 2.0 g mineral sample was loaded into a 30 mL flotation tank, and 20 mL of deionized water was then added. After the pH value was adjusted to a required pH value with the pH regulator for 1 min, the activator, depressor, and collector were then added in order. The pulp was agitated for 3 min after each reagent was added, and the floated materials were collected for 3 min. Foam products and tank minerals were obtained by filtration, and they were dried and weighed to calculate the recovery.

2.3. Zeta potential measurements

Zeta potentials were measured by a Malvern Instruments Nano-ZS90 zeta potential analyzer. The sample was finely ground to −5 μm powder in an agate mortar. The powdered mineral was weighed into beakers (20 mg each) and added to 50 mL of deionized water. The pulp pH value was regulated with 0.10 mol/L HCl or 0.10 mol/L NaOH solutions. The reagents were then added, followed by 5 min of stirring with a magnetic stirrer. A moderate amount of pulp was injected through a syringe into the test electrophoresis tube for zeta potential measurement.

2.4. Adsorption tests

The adsorption tests were performed in Spectro Flex 6600. 2 g of mineral samples were taken and made up to

100 mL after addition of a desired concentration of regents in 250 mL Erlenmeyer flasks. The suspensions were mixed and placed on a rotator for 1 h to ensure that the adsorption process had reached equilibrium. At this equilibrium point, we assume that the depleted dosage of reagent had been adsorbed onto the mineral surface. Finally, the mineral solution was centrifuged, and the supernatant was used to measure the residual reagent concentration.

2.5. FTIR spectroscopy

The mineral samples were finely ground to -2 µm in an agate mortar. Then, 2-g mineral samples were added to a 50-mL aqueous solution in the absence and presence of reagents. After being stirred for 30 min, the minerals were filtered and washed with ultra-pure water three times and, subsequently, dried in a vacuum oven at 40°C for 24 h. The analyses were performed by Nicolet 380 FTIR spectrometer using potassium bromide pellets, which were pressed by mixing 1 mg sample and 100 mg KBr.

3. Results and discussion

3.1. Effects of siderite on mixed minerals (quartzhematite) flotation

Flotation of three single minerals, namely hematite, siderite, and quartz, in an oleate-starch-CaCl₂ system at different pH values was examined. The corresponding results are shown in Fig. 1. Sodium oleate and CaCl₂ was used as a collector and an activator for quartz, respectively. Starch was used for the depressant of iron minerals (hematite and siderite). It is shown that the floatability of quartz and iron minerals is extremely different in the oleate-starch-CaCl₂ system when the pH value is 11, which indicates that separation of quartz and iron minerals is feasible.

Fig. 1. Flotation recoveries of siderite, hematite, and quartz as a function of pH values in the presence of 160 mg/L sodium oleate, 60 mg/L starch, and 100 mg/L CaCl₂.

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The effect of siderite on flotation of mixed mineral samples at a pH value of 11 is shown in Fig. 2. The samples were made up of hematite, siderite, and quartz at different mass ratios. Here the mass ratio of hematite to quartz equals one, and the content of siderite was calculated on the basis of the mineral composition. It can be seen that the iron grade decreases significantly with an increase of siderite content in the mixtures. When there is no siderite in the mixed samples, the iron grade is 62.1%. The iron grade decreases to 38.3% when the siderite content is increased to 20%. Though the addition of siderite to the mixture might slightly increase the reagent consumption and potentially affect the flotation results, the effect of siderite on separation of quartz from hematite is much more significant than what was expected. When the content of siderite is high, separation of quartz and hematite is very difficult. The mechanism of siderite effects will be discussed in the following experiments.

Fig. 2. Effect of siderite on the grade of iron concentrate (sodium oleate, 160 mg/L; starch, 60 mg/L; CaCl₂, 100 mg/L).

3.2. Effects of siderite on single mineral flotation

The effects of siderite on flotation of hematite and quartz in the oleate-starch-CaCl₂ system were investigated further. To clarify the effects of slime adhesion and dissolved species, siderite mixed with quartz or hematite was classified into coarse $(-106 + 45 \text{ µm})$, fine (-18 µm) , and coarse–fine (the mass ratio of coarse to fine particles equals one). Coarse siderite was directly mixed with other minerals, whereas fine siderite was stirred for 30 min to decant solution of dissolved species before mixing with quartz or hematite. Coarse particles and fine particles in the coarse–fine siderite were treated along the above requirements.

The effects of siderite on flotation of hematite are lower than those on quartz as shown in Fig. 3 and Fig. 4. The hematite that was mixed with different types of siderite is almost effectively depressed, and siderite barely influences hematite floatability in the flotation system. However, siderite could significantly affect the flotation of quartz according to the results of Fig. 4, which indicates that the effects of siderite on the reverse flotation of hematite are mainly realized by influencing quartz floatability. The recovery of quartz reduces from 90.05% to 60.06% when the fine siderite mass ratio increases from 0 to 10%, revealing that the fine siderite particles partly influence flotation of quartz. When the mass ratio of coarse siderite increases from 0% to 10%, the recovery of quartz reduces from 90.05% to 15.32%. Compared with the fine siderite, it can be concluded that the dissolved species could significantly affect flotation of quartz assuming that coarse siderite particles barely adhere on quartz surfaces. Compared with fine siderite (coarse siderite), the recovery of quartz that was mixed with the coarse–fine siderite decreases from 60.06% (15.32%) to 8.56% when the mass ratio of siderite is 10%; it indicates that the synergistic effects of fine particles and dissolved species could enhance interactions between quartz and siderite.

Fig. 3. Effects of siderite on flotation of hematite (pH value, 11; sodium oleate, 160 mg/L; starch, 60 mg/L; CaCl₂, 100 **mg/L).**

Fig. 4. Effects of siderite on flotation of quartz (pH value, 11; sodium oleate, 160 mg/L; starch, 60 mg/L; CaCl₂, 100 mg/L).

The abovementioned results show that interactions between quartz and siderite are the main reasons why siderite significantly influences reverse flotation of hematite. The

interactions are related to the dissolved species and fine particles, and the dissolved species dominate in the interactions. The mechanism needs to be analyzed by further tests and theoretical calculations.

3.3. Adsorption tests and FTIR spectroscopy

In general, the recovery of minerals is related to the adsorption quantity of the collector. Investigating the adsorption behavior contributes to the analysis of the mechanism of interactions between quartz and siderite. The effects of siderite on the adsorption quantity of sodium oleate on quartz were studied by adsorption tests, and the corresponding results are shown in Fig. 5. Fine siderite that was mixed with quartz underwent the same pretreatment as in single mineral flotation. According to the results, the combined action of coarse siderite and starch heavily reduces the adsorption quantity of quartz, whereas coarse siderite slightly brings adsorption quantity down in the absence of starch. The effect of fine siderite on the adsorption quantity is hardly connected with starch, which partly reduces the adsorption quantity. The adsorption test results indicate that starch can enhance the effect of coarse siderite (dissolved species), not fine siderite, on quartz floatability.

Fig. 5. Adsorption quantity of sodium oleate on quartz (pH value, 11; sodium oleate, 160 mg/L; CaCl₂, 100 mg/L; mass ra**tio of siderite, 10%).**

To further clarify the mechanism of the combined action of starch and coarse siderite (dissolved species), the FTIR spectra of quartz interacting with starch was performed. Moreover, the result is shown in Fig. 6. Characteristic peaks at 3424 cm⁻¹ and 1620 cm⁻¹ (curve 1) are due to the stretching vibration and bending vibration of –OH from Si–OH silanol groups in defect sites [15−16]. A peak located at the wavenumber of 1081 cm^{-1} is due to Si–O–Si asymmetric stretching vibration that is the first characteristic absorption peak of quartz [17−18]. A comparison of curves 1 and 2 shows that neither new peaks found nor translation peaks

are formed, indicating that starch hardly adsorbs on quartz surfaces. A new peak appeared at 1404 cm^{-1} (curve 3), which represents the bending vibration of –CH– from starch [19−20]. Characteristic peaks due to the stretching vibration and bending vibration of –OH from Si–OH silanol groups are shifted by approximately 23 cm⁻¹ (from 3424 cm⁻¹) and 11 cm^{-1} (from 1620 cm⁻¹) to low frequencies, indicating that –OH is involved in the adsorption process and that hydrogen bonding adsorption occurs.

Fig. 6. FTIR spectra of quartz interacting with starch: $1 -$ **quartz;** $2 -$ **quratz** + CaCl₂ + starch; $3 -$ **quartz** + CaCl₂ + **starch + siderite saturated solution.**

The results of adsorption tests and FTIR spectroscopy show that fine siderite particles influence quartz flotation by directly reducing the adsorption quantity of the collector, whereas the dissolved species, by enhancing starch interaction with quartz, significantly prevent collector adsorption.

3.4. DLVO theoretical calculations

The results of single mineral flotation and adsorption tests show that fine siderite particles partly influence flotation of quartz. This influence may be due to fine particles adhesion. The mechanism is analyzed on the basis of DLVO theory calculations. According to the DLVO theory, repulsion and agglomeration between particles are mainly determined by their electrostatic forces and Vander Waals forces. The total interaction energy is given by

$$
V_{\rm T} = V_{\rm W} + V_{\rm E} \tag{1}
$$

where V_T , V_W , and V_E are the total interaction energy, Van der Waals energy, and electrostatic interaction energy, respectively. The Van der Waals energy of two spherical particles can be calculated as

$$
V_{\rm w} = -\frac{AR_{1}R_{2}}{6H\left(R_{1} + R_{2}\right)}
$$
 (2)

where $A = (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}})$, *H* is the distance between two mineral particles, *R* is the radius of spherical

particles, A_{11} is the Hamaker constant of mineral 1, A_{22} is the Hamaker constant of mineral 2, and A_{33} is the Hamaker constant of water in vacuum. The Hamaker constant of quartz, siderite, and water are 5.0×10^{-20} , 18.0×10^{-20} , and $4 \times$ 10^{-20} J, respectively. The electrostatic interaction energy of two spherical particles is usually determined by the following equation:

$$
V_{\rm E} = \frac{\pi \varepsilon_0 \varepsilon R_1 R_2}{R_1 + R_2} \left(\varphi_{01}^2 + \varphi_{02}^2 \right) \left(\frac{2 \varphi_{01} \varphi_{02}}{\varphi_{01}^2 + \varphi_{02}^2} p + q \right)
$$
(3)

$$
p = \ln \frac{I + \exp(-\kappa H)}{I - \exp(-\kappa H)},
$$

$$
q = \ln [1 - \exp(-2\kappa H)].
$$

Here *κ* is the Debye length and is 0.104 nm⁻¹; ε_0 and ε are the permittivity of vacuum and of the solution (water is 78.5 $C^2 \cdot J^{-1} \cdot m^{-1}$), respectively; φ_{01} and φ_{02} are the surface potential of quartz and siderite, respectively.

The interaction energy of siderite particles, of different sizes, with quartz was calculated according to Eqs. (1) – (3) and the result is shown in Fig. 7. All curves were carried out in the oleate-starch-CaCl₂ solution and the surface potential of siderite and quartz is -30 and -35 mV, respectively. It can be seen that siderite particles could interface with quartz when the value of *H* is less than 2.5 nm. The value of V_T increases as the particle size increases when the value of *H* is more than 2.5 nm, which indicates that fine siderite particles adhere on quartz more easily compared with the coarser ones. The results confirm that the adhesion increases when siderite particle size decreases. Siderite slime (fine particles) might influence metal ions (Ca^{2+}) adsorption on quartz surfaces by adhesion or by covering the metal ions present on the surfaces, reducing the active adsorption sites of quartz and at the same time the adsorption quantity of the collector on quartz. This result is in good agreement with the literature [9−10].

Fig. 7. Total interaction energy between siderite particles of different sizes and quartz.

3.5. Chemical solution calculations

The results of FTIR spectroscopy show that the dissolved species of siderite enhance starch interaction with quartz, an indication that the surface of active quartz might be converted to something depressed by starch. Chemical solution calculations that play a major role in investigating the behavior of the flotation system are used to analyze the effect of dissolved siderite species on quartz flotation. The formation of a particular mineral in a solution containing A^{n+} and B^{*m*−} can be expressed by the following equation:

$$
A_{m}B_{n}(s) = mA^{n+}(aq) + nB^{m-}(aq), K_{sp}
$$
 (4)

where $K_{\rm sp}$ is the corresponding solubility product constant. Due to the requirements of chemical solution calculations, the ions activity (IA) and saturation index (SI) are given by

$$
IA = \left[A^{n+}\right]^{m} \left[B^{m-}\right]^{n}; SI = \lg IA - \lg K_{sp} \tag{5}
$$

Bulk precipitation of the mineral is anticipated when $IA > K_{sp}$ (SI > 0) according to the principles of solution chemistry, where $[A^{n+}]$ and $[B^{m-}]$ are the actual activities of A^{n+} and B^{n+} in solution, respectively. A value greater than zero indicates that the bulk precipitates are anticipated to be formed, whereas a negative value indicates that the precipitates may be dissolved.

The conditioning time is limited in flotation, but mineral suspensions may require several hours or weeks for the equilibrium of absorption of $CO₂$ into carbonate solutions [21−22]. Especially at the alkaline region, the increase of bicarbonate concentration in the solution makes adsorption of $CO₂$ more difficult. Therefore, the chemical solution calculations are suitable for the closed system. The CaCl₂− NaOH−siderite−quartz system is used to investigate the effect of dissolved species on flotation of quartz. The corresponding reactions and constants for the chemical solution calculations are shown in Table 1. At the alkaline region, equations based on the solubility equilibrium, charge conservation, and proton conservation are listed below:

$$
\begin{cases}\n[CO_3^{2-}][Fe^{2+}] = K_{\rm spl} \\
[Ca^{2+}]_T = [Ca^{2+}] + [CaOH^+] + [Ca(OH)_2] \\
[H^+] + 2[Fe^{2+}] + [FeOH^+] + 2[Ca^{2+}] + [Na^+] + \\
[CaOH^+] = [OH^-] + [Cl^-] + \\
2[CO_3^{2-}] + [HCO_3^-] + [Fe(OH)_3^-] + 2[Fe(OH)_4^{2-}] \n\end{cases}
$$
\n
$$
[H^+] + [HCO_3^-] + 2[H_2CO_3] = [OH^-] - [Na^+] + \\
[CaOH^+] + 2[Ca(OH)_2] + [Fe(OH)^+] + \\
2[Fe(OH)_2] + 3[Fe(OH)_3^-] + 4[Fe(OH)_4^{2-}]
$$

To reduce the number of indeterminate ions, the unknowns are set as follows:

$$
\left[\text{Ca}^{2+}\right] = x; \left[\text{Fe}^{2+}\right] = y; \left[\text{CO}^{2-}_3\right] = z; \left[\text{Na}^+\right] = m \tag{7}
$$

The concentration of Cl[−] is considered to be 2.0×10^{-3} mol/L (the dosage of CaCl₂ reaches 100 mg/L), which is convenient for chemical solution calculations. According to the reactions and the constants in Table 1, Eq. (6) can be translated into the following:

$$
\begin{cases}\nyz-10^{-10.68} = 0 \\
x+10^{pH-12.6} x+10^{2pH-25.23} x-10^{-3} = 0 \\
10^{-pH} -10^{pH-14} + 2x+10^{pH-12.6} x+2y+10^{pH-9.5} y - \\
10^{3pH-32} y - 2 \times 10^{4pH-46.4} y - \\
2z-10^{10.33-pH} z + m - 2 \times 10^{-3} = 0 \\
10^{-pH} -10^{pH-14} -10^{pH-12.6} x - 2 \times 10^{2pH-25.23} x - \\
10^{pH-9.5} y - 2 \times 10^{2pH-20.6} y - 3 \times 10^{3pH-32} y - \\
4 \times 10^{4pH-46.4} y + 10^{10.33-pH} z + 2 \times 10^{16.68-2pH} z + m = 0\n\end{cases}
$$
\n(8)

In the $CaCl₂-NaOH-siderite–quartz system, the concen$ tration of $\lceil Ca^{2+} \rceil_T$ that is used as an activator for quartz is relatively high. We anticipate that the dissolved species of siderite may convert active constituents $(Ca(OH))$ ⁺ and $Ca(OH₂)$ of quartz to $CaCO₃$ precipitates. To investigate whether $CaCO₃$ precipitates will form or not, the saturation index of $CaCO₃$ is taken as the criterion. The value of SI can be solved by Eq. (8) and turned into the following:

$$
\begin{array}{l}\n\left.S\right]_{CaCO_3} = \lg\left[\frac{10^{-3}}{K_{sp}\left(1+10^{pt-12.6}+10^{2pt-25.23}\right)}\right.\n\left.\left.\sqrt{\frac{10^{-10.68}\left(1+10^{pt-9.5}+10^{2pt-20.6}+10^{3pt-32}+10^{4pt-46.4}\right)}{1+10^{10.33-pH}+10^{16.68-2pt}}\right]}\n\end{array}\n\right]
$$
\n(9)

Table 1. Chemical reaction equations and equilibrium constant for the chemical solution calculations

Reaction	Equilibrium constant
$H2O \rightleftarrows H++OH-$	$K_{\rm w} = 10^{-14}$
$\text{FeCO}_{3(s)} \rightleftarrows \text{Fe}^{2+}+\text{CO}_3^{2-}$	$K_{\rm{spl}} = 10^{-10.68}$
$CaCO3(s) \rightleftarrows Ca2+ + CO32-$	$K_{sp2} = 10^{-8.35}$
$CO_3^{2-}+H^+ \rightleftarrows HCO_3^-$	$K_1 = 10^{10.33}$
$HCO32-+H+ \rightleftarrows H, CO3$	$K_2 = 10^{6.35}$
$Fe^{2+}+OH^{-} \rightleftarrows FeOH^{+}$	$\beta_1 = 10^{4.5}$
$Fe^{2+}+2OH^{-} \rightleftarrows Fe(OH)$,	$\beta_2 = 10^{7.4}$
$Fe^{2+}+3OH^{-} \rightleftarrows Fe(OH)_{2}^{-}$	$\beta_3 = 10^{10.0}$
Fe^{2+} +4OH ⁻ \rightleftarrows Fe(OH) ²⁻	$\beta_4 = 10^{9.6}$
$Ca^{2+}+OH^{-} \rightleftarrows CaOH^{+}$	$\beta_5 = 10^{1.4}$
$Ca^{2+}+2OH^{-} \rightleftarrows Ca(OH)$,	$\beta_{6} = 10^{2.77}$

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The saturation index of $CaCO₃$ is a function of pH values according to Eq. (9) and the relationship with pH values is shown in Fig. 8. The value of SI is above zero when the pH value is greater than 9.9, indicating that $CaCO₃$ precipitates are anticipated to be formed at the same range of pH values. The adsorption quantity of Ca^{2+} on quartz increases obviously when the pH value is higher than 10, suggesting that CaCO₃ precipitates are mainly formed on the surface of quartz [23−24]. The dissolved species of siderite is complex, it contains, in fact, carbonate ions, ferrous ions, and their hydrolysis ions. The results of chemical solution calculations show that ferrous ions and its hydrolysis ions are trace amounts compared with calcium concentration. Therefore, the effects of ferrous ions (or its hydrolysis ions) can be ignored. Therefore, it can be concluded that the dissolved siderite species changes the surface of active quartz to CaCO₃ precipitates that can be depressed by starch, and this explains how the dissolved siderite species can enhance starch interaction with quartz. $CaCO₃$ precipitates on quartz interact with sodium oleate less strongly when compared with hydroxylate surfaces $(Ca(OH)^+$ and $Ca(OH)_2)$, which might be the reason that the dissolved species of siderite could slightly reduce the adsorbed quantity of sodium oleate on quartz in the absence of starch. The results of chemical solution calculations agree with those of FTIR spectroscopy and adsorption tests.

Fig. 8. Relationship between pH values and saturation index.

4. Conclusions

(1) Micro flotation results show that interactions between siderite and quartz are the main reasons that siderite significantly influences flotation. The interactions are related to the dissolved siderite species and fine siderite particles, and the dissolved species dominate in the interactions.

(2) Adsorption tests and FTIR spectroscopy indicate that

fine siderite particles influence quartz flotation by directly reducing the adsorption quantity of the collector, whereas the dissolved species, by enhancing starch interaction with quartz, significantly prevents collector adsorption.

(3) The effects of siderite on flotation of quartz are realized by slime adhesion and surface conversion. DLVO theoretical calculations confirm that fine siderite particles adhere on quartz more easily compared with coarser particles. Chemical solution calculations indicate that the dissolved species of siderite might change the surface of active quartz to $CaCO₃$ precipitates that can be depressed by starch. The theoretical calculations are in agreement with the results of adsorption tests and FTIR spectroscopy.

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