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Preparation of High Purity Boric Acid by Modified Peanut Shells

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Abstract

the modification of peanut shell by nitric acid, sodium hydroxide, and epichlorohydrin have been conducted accordingly, and application in the purification of high purity boric acid from industry products have been carry out, the result indicated that modification by epichlorohydrin was more effective than by nitric acid and by sodium hydroxide in the purification which the iron ion, sulfate ion, and chloridion can be removed simultaneously from industry boric acid, the analytical result by IR, SEM, and XRD confirmed that there are amount of hydroxyl and carboxyl group and ether bound in the modified peanut shell as functional group for adsorption of various metals ion and anions.

Keywords: peanut shell; modification; adsorption; boric acid.

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Introduction

Peanut shell as a by-product of peanut are low in density and high in volume and are used in animal feed or burned for energy, in order to add value to peanut shells, attention has focused on the utilization of peanut shells as adsorbents after modification by physics and chemistry treatment, there have been several reports about peanut shell for adsorption of various metals ion or organic compounds from solution[1-7] and for filter or reinforcement of materials[8-10]. The kinetics, isotherms and thermodynamics about the adsorption of metal ion from solution were investigated[11-13].There are a lot of cellulose, hemicelluloses, lignin et. al. in the composition of original peanut shell, and the functional group in them which including hydroxyl, amino, carboxylic and phenolic hydroxyl groups et. al. have the ability of assembling with metals ion, the modification mechanism was uncertain, but adjusting the functional group of hydroxyl, carboxylic , and surface electronic properties by acidic, alkali, et.al. chemical treatment have been understanding[1,2,4,6,11]. Modification of peanut shell by sodium hydroxyl and epichlorohydrin indicated that the group of carbonyl was removed, hemicelluloses and lignin was dissolved, cellulose has been run and the specific surface area expanded, and the C-O bound and epoxy groups have been increased as a result of graft reaction taken place between the epichlorohydrin and group of hydroxyl of peanut shell[1,11,14].In order to got the high purity boric acid, recrystallization, together assembling, adsorption with recrystallization from industry boric acid are general method[15,16], the removal of ion of above method was only single, but the standard concentration of high purity boric acid include negative ions such as sulfate ion, chloridion, or phisphloc, metals ion including heavy metal, iron ion, and some undissolved materials in water. The purpose of this study proved that the peanut shell modified by sodium and epichlorohydrin was an effective adsorbent for purification of industry boric acid to got the high purity boric acid, it can remove the sulfate ion, chloridion, iron ion and some undissolved material in water of industry boric acid simultanously.

Experimental

Peanut shell, from countryside of JINZHOU city, industry boric acid from plant in KUANDIAN county, the epichlorohydrin, nitric acid, and sodium hydroxide was analytically pure, sulfate ion, iron ion was determined according to the GB-T12684-2006(China), and the concentration of chloridion determined by spectrophotometry, epoxide number was determined by Hydrochloric acid-acetone method.

Peanut shell was washed and dried firstly, 20-40 mesh of powder was obtained by grinding sieving,

10g of the powder and 125ml, 10%(v), nitric acid was put into the 250-cc round-bottomed flask respectively, reaction for 3 hours at 80℃ with stirring, the residue was washed by distilled water until neutral, drying the standby at 40℃.

10g of the powder and 100ml, 1.5mol/L, sodium hydroxide was put into the 250-cc round-bottomed flask respectively, reaction for 40minutes at 30℃ with stirring, the residue was washed by distilled water until neutral, drying the standby at 50℃.

10g of the powder, 100ml, 1.5mol/L, sodium hydroxide, and 15ml, epichlorohydrin was put into the 250-cc round-bottomed flask respectively, reaction for 40minutes at 30℃ with stirring, the residue was washed by distilled water until neutral, drying the standby at 50℃.

20g of industry boric acid was dissolved in 300ml of water, a balanced amount of modified peanut shell was putted into the solution and used as the absorbents, static adsorption for a certain temperature, separated the peanut powder from solution, and purred boric acid obtained by recrystallization.

Result and discussion

The removal rate of sulfate ion, chloridion and iron

The removal rate of sulfate ion, chloridion and iron with adsorption time by nitic acid, sodium hydroxide and epichlorohydrin were shown in figure1, figure2, and figure3 respectively, it can be seen that the removal rate of sulfate ion by alkali-activated, and graft modification was more than by acid modification, but there was an adsorption equilibrium after adsorption for 20 minutes in figure1. The result of adsorption for chloridion was roughly the same in figure2. And there was an effective removal of iron ion by graft modification, the removal of by acid modification was effective only at firstly, and general effective for alkali-activated in figure3. The result indicated that graft modification of peanut shell for removal of sulfate ion, chloridion and iron ion was effective simultaneously.

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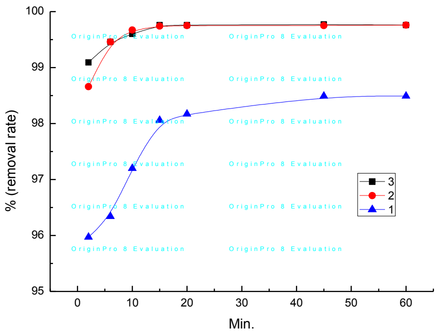

Figure1 The removal rate of sulfate ion vs. adsorption time(1: acid, 2: alkali, 3: graft)

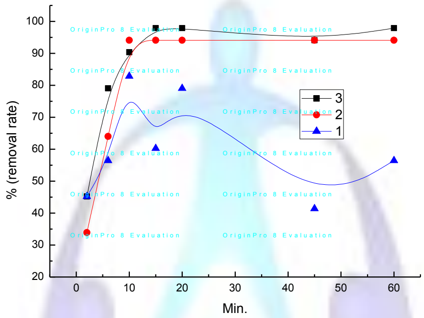

Figure2 The removal rate of chloridion vs. adsorption time (1: acid, 2: alkali, 3: graft)

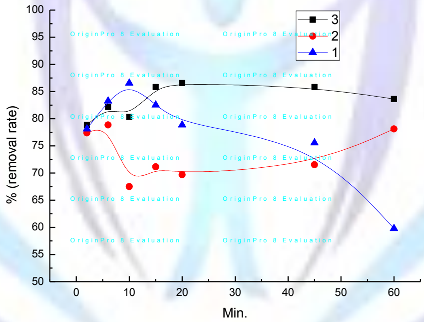

Figure3 The removal rate of iron ion vs. adsorption time (1: acid, 2: alkali, 3: graft)

The result of super purified boric acid

The result of super purified boric acid was shown in table 1. It can be seen that the content of foreign ion include sulfate ion, chloridion, iron, and water insolubles was reached the high quality (ppm class) .

Table 1 The result of super purified boric acid

Boric acid	Content（%）				
	SO_4^{2-}	Cl^-	Fe^{3+}	water insoluble	total
Industrial	0.3100	0.0567	0.0065	0.0864	99.5
Prepared	0.0008	0.0015	0.0009	0.0018	99.99

IR pattern of modified peanut shell and after adsorption

IR pattern of modified peanut shell and after adsorption was shown in figure4 and figure5, it can be seen that the absorption peaks at 3418cm-1, 2922cm-1, 1423cm-1, 1374cm-1, and 894cm-1 belong to the characteristic absorption peaks of the cellulose, 1735cm-1 of hemicelluloses, 1634cm-1, and 1511cm-1 of lignin for original peanut shell in figure4 a, the weaken of the absorption peaks at 894cm-1, 1056cm-1, 1511cm-1, and 374cm-1 confirmed that the cellulose, hemicelluloses, and lignin in original peanut shell was involved in the modified reaction, alkaline degradation, pealing reaction, and acetyl fall off taken place as a result of breakage of hemicellulose glucoside bounds under the condition of acid, but functional groups of hydroxyl and amino was blocked by the increased surface electronegativity, so as to the acidic including carboxylic and phenolic hydroxyl groups was protected, and this help with cation exchange and complexation.

There was no characteristic absorption peaks of 1735cm-1 of hemicelluloses, and marked change at 1634cm-1 by alkali-activated, the result indicated that the cellulose has been run and the specific surface area expanded, it make more adsorption functional groups in cellulose leaked and naked. The weaken of characteristic absorption peaks at 899cm-1 forβ-glycosidic bond, 1265cm-1 for CH2-OH, 1511cm-1 and 1634cm-1 for lignin confirmed that alkaline hydrolysis taken place in lignin, the functional groups of hydroxyl and amino was protected and carboxylic and phenolic hydroxyl groups was blocked when the peanut shell treated by alkali-activated.

The distinct weakening at 3418cm-1by graft modification than alkali-activated indicated that groups of hydroxyl reacted with epichlorohydrin, and it was stronger at 1265cm-1 than alkali-activated indicated that C-O bound was increased, and the weaken of 1280cm-1 to 1180cm-1 indicated that the epoxy groups was in open loop, and cross-linking reaction was taken place by graft modification combined with the determined result of epoxide number of 0.1163 more than 0.0198 in original peanut shell.

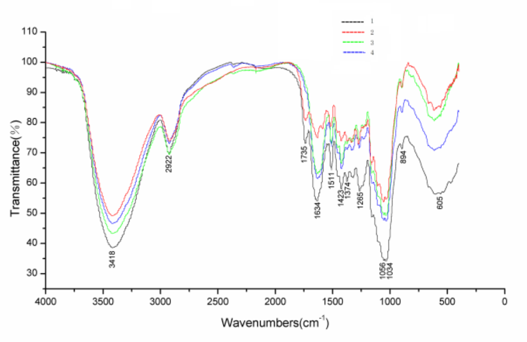

Figure4 IR pattern of modified peanut shell (1: original, 2;acid, 3: alkali,4: graft)

The weaken of absorption peaks at 3418cm-1 and 1734cm-1 confirmed that the hydroxyl and carboxyl group was the functional group in adsorption by acid modification, the weaken of absorption peaks at 1056cm-1 and 1034cm-1 indicated that it lead to further lignin dissolved in acidic conditions.

The stronger of absorption peaks at 3418cm-1 indicated that the increased hydroxyl group improve the effect of the adsorption of anionic, combined with that adsorption effect of iron ion becomes poor by alkali-activated, the carboxyl group was the functional group in adsorption of ion, and hydroxyl group is also effective for adsorption of iron ion.

IR pattern after adsorption by graft modification was similar to by alkali-activated, the ether bond and hydroxyl group have been increased by graft modification than by alkali-activated, the more effective adsorption for ion.

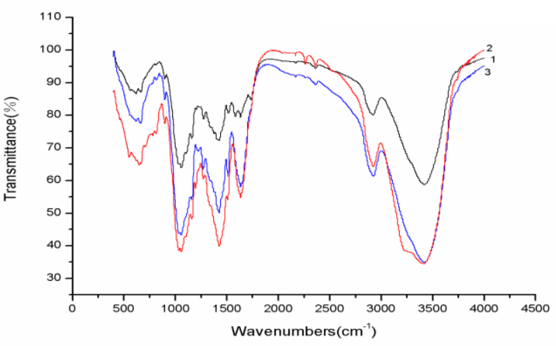

Figure5 IR pattern of modified peanut shell after adsorption (1:acid, 2: alkali,3: graft)

SEM image of peanut shell

SEM image of original, modified and adsorption peanut shell was shown in figure6 (a, b, c, d, and e, f, g), it can be seen that there are uneven surface, aggregation, and a lot of holes in the original peanut shell in figure6 a, there were flat surface, less holes in the acid modification peanut shell in figure 6 b and obvious shrinkage after adsorption in figure 6 e, the alkali-activated result is just the opposite to acid modification, the result indicated that the microstructure of different as a result of different modified functional group by acid modification and alkali-activated. There are dendritic structure on the surface, obviously aggregation, uneven surface by graft modification, and the disappearance of the structure after adsorption confirmed that the adsorption was highlight on the dendritic structure.

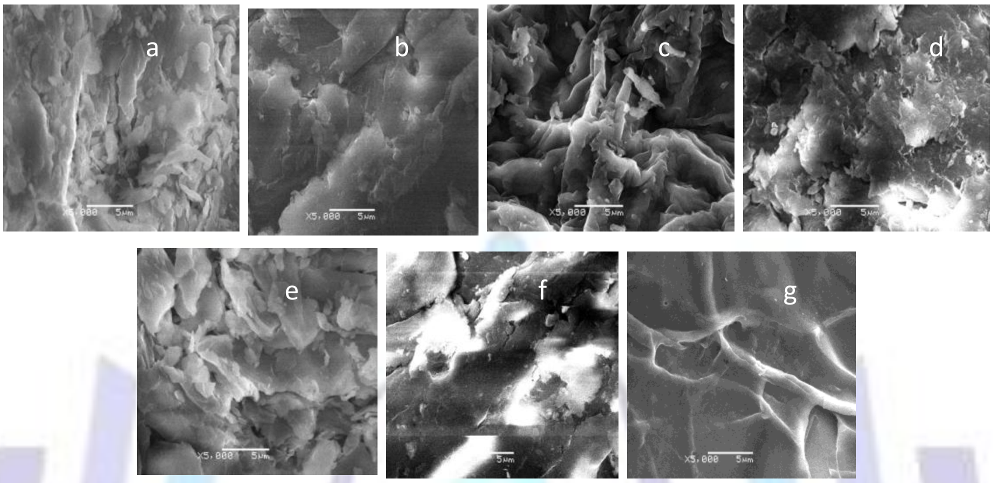

Figure6 SEM image of original, modified and adsorption peanut shell (modification:a:original, b:acid, c:alkali, d:draft; after absorption: e:acid, f:alkali, g;graft)

XRD pattern of modified peanut shell and after adsorption

XRD pattern of modified peanut shell and after adsorption was shown in figure7 (a, b), the peaks as 2θ at 16°and 22°was the characteristic peak of cellulose, 22°was the mark of cellulose high crystallization, and 16°was the oligosaccharide structure of low crystallinity in the figure 7(a), but the characteristic peak at 28°indicated that the adsorption properties of peanut shell by graft modification was better than by acid modification or alkali-activated in the figure7(b).

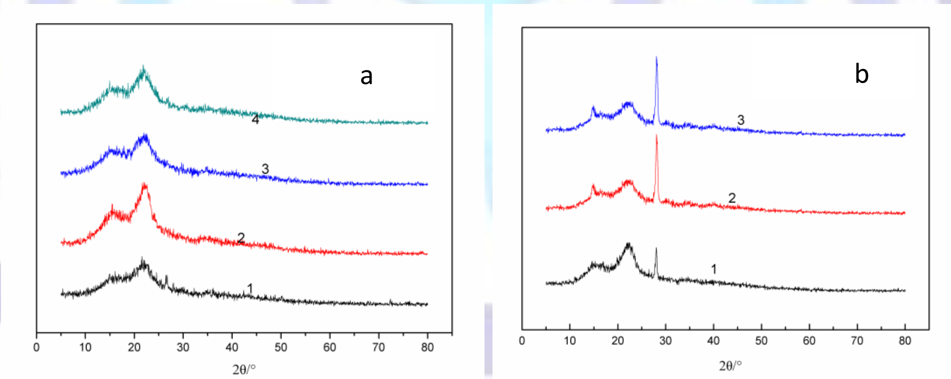

Figure7 XRD pattern of modified peanut shell and after adsorption (a:before, 1:original, 2:acid, 3: alkali, 4:graft; b:after adsorption, 1, acid, 2: alkali, 3:graft)

Conclusion

The graft modification of peanut shell for removal of sulfate ion, chloridion and iron ion was effective simultaneously, there was an adsorption equilibrium after adsorption for 20 minutes. the content of foreign ion included sulfate ion, chloridion, iron, and water insolubles was reached the high quality (ppm class) . the ether bond and hydroxyl group have been increased by graft modification , the hydroxyl and carboxyl group was the functional group in adsorption of iron ion., the carboxyl group was the functional group in adsorption of ion, There are dendritic structure on the surface, obviously aggregation, uneven surface by graft modification, and the disappearance of the structure after adsorption confirmed that the adsorption was highlight on the dendritic structure.

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