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PREPARATION AND CHARACTERIZATION OF ALUMINUM STEARATE

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Preparation of aluminum stearate by the precipitation method was examined under various conditions of stearic acid saponification with sodium hydroxide. It was proved that the most favorable ratio of acid/alkali was 1:1.5 and that the obtained soap was very similar to the commercial product. Endothermic effects determined by differential scanning calorimetry and also the other parameters showed that the soaps consisted mono-, di-, tristearates and non-reacted substances, where distearate was the dominant form.

KEY WORDS: aluminum stearate, soap, characterization

INTRODUCTION

Aluminum soaps have different and widespread applications (1-4), aluminum stearates being among them especially important. Aluminum tristearate is useful in paints and enamels and in the flatting of varnishes. It also prevents bleeding and oil separation in putty. Aluminum distearate is the most commonly used grade of aluminum stearate. It is used as a thickener in paints, inks and greases; a water repellent for leather and rope; and a lubricant in plastics and rope. It is also used in cement production for waterproofing and air entrainment, and in hot-melt paper coating compounds. Because of its unusually heavy bodying properties, aluminum monostearate is used in the manufacture of paints, inks, greases, and waxes (3). In recent years (5), hydrophobic aluminum monostearate was tested as a low-solubility denitrification substrate for anaerobic bacteria and a source of aluminum for phosphate precipitation. Aluminum stearate has potential for use in a flow-through container for denitrification of oxidized effluent from home sewage systems. It was also referred that the preheated mixture of metal soaps, along with the costabilizer, delayed the fast blackening of the polymer, but mainly showed a reduction

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in the rate of dehydrochlorination, which subsequently produced a less black material (6). Stearic acid saponification conditions are very important for the process of obtaining mono-, di- and tristearate aluminum soaps. For this process is also important the amount of applied alkali, i.e. molar ratio of the acid and alkali. In the saponification reaction

$$CH_3(CH_2)_{16}COOH + n\ NaOH \rightarrow CH_3(CH_2)_{16}COONa + H_2O + (n-1)\ NaOH$$

n influences the equilibrium. When n value is higher, the reaction equilibrium is shifted to the right and a smaller amount of free stearic acid remains as unreacted in the soap (7).

Manufacture of mono-, di- and tristearate aluminum soaps is mainly empirical. Important preparation parameters are the amount of NaOH for saponification, temperature of saponification and precipitation, mixing speed, precipitation speed, etc.

In this study, aluminum stearate precipitation was carried out with different molar ratio of reactants. The objective was to investigate optimal ratio of reactants and characterize the obtained soaps.

EXPERIMENTAL

Procedure of aluminum stearate precipitation

Aluminum soaps were obtained by precipitation from sodium stearate (pH<10.5) with aluminum(III)-chloride solution. The ratio of sodium stearate and aluminum(III) chloride was 1:1.5.

The obtained precipitate was separated by filtering on a Büchner funnel, washed with warm water and dried in laboratory and vacuum drier to the constant mass (8).

Water-soluble sodium stearate has been prepared by direct saponification of stearic acid at 55°C (9-12) with the ratios of stearic acid and sodium hydroxide: 1:1.2; 1:1.4; 1:1.5.

Characterization of aluminum stearate

Melting point: Method by Koffler (Franz Küstner, Dresden, Germany) and differential scanning calorimetry, DSC (910 Differential Scanning Calorimeter, Du Pont Instruments, USA). Heating speed of samples was 10°C/min.

Ash: Total ash and insoluble ash (13)

Aluminum content: Complexometry (14).

Free fatty acids (FFA): extraction with diethyl ether (15).

Density: determined in benzene with Gay-Lussac picnometer (16).

For comparison of the obtained results commercial aluminum stearate C.12 (“Lek”, Ljubljana, Slovenia) was used.

All chemicals were of analytical reagent grade (p.a.).

Stearic acid was puriss grade.

RESULTS AND DISCUSSION

Comparison of the literature data (1, 6, 17) and obtained results showed a good agreement.

Results are presented in Table 1 and Fig. 1.

Table 1. Characteristics of aluminum stearate samples obtained by the reaction of different stearic acid (SA) and sodium hydroxide (NaOH) molar ratios

Characteristics	SA/NaOH ratio			
	1:1.2	1:1.4	1:1.5	C.12*
MP** by Koffler (°C)	122-128	140-151	138-143	144-152
MP by DSC (°C)	71, 90	74, 90, 135, 181	75.5, 90, 127, 157	55, 86, 120, 146
Total ash (%Al)	3.74	3.86	3.97	5.16
Water-insoluble ash (%Al)	1.60	1.78	1.83	2.12
Aluminum content (%)	1.32	1.62	1.91	2.01
Free fatty acids (%)	15.86	7.60	4.41	8.22
Density (g/cm^3)	0.9691	0.9298	0.9623	0.9943

* MP-melting point
** C.12-commercial aluminum stearate

Melting points of soaps determinated by the Koffler method showed that decrease of the acid/alkali ratio resulting in lowering of the melting point of the soap (Table 1). Since DSC method is more sensitive and more appropriate for this kind of samples, the corresponding DSC curves were recorded. DSC analysis of the obtained soaps showed several endothermic effects that are more or less expressed (Fig. 1).

Several peaks for one soap curve suggest that the given soap sample was not pure, i.e. it represented a mixture of different acids or of different salt forms and also a combination of the both. Comparison of DSC curves of different aluminum soaps precipitated with different acid/alkali ratios indicated that the maxima of endothermic effects were shifted to lower temperatures. This means that the obtained soaps contained free reactants. Theese endothermic effects were less pronounced at higher acid/alkali ratio. The peaks at temperatures of 55°C, 86°C, 120°C and 146°C on the DSC curve of commercial aluminum stearate, represent melting points of stearic acid, free reactants, aluminum tri-/di-stearates and aluminum distearate, i.e. overall aluminum stearate, respectively (2, 3, 17). The most similar to aluminum stearate commercial soap is one precipitated with the acid /alkali ratio 1:1.5. The endothermic effect temperatures at 75.5°C, 90°C, 127°C and 157°C indicated that the precipitated aluminum soap contained non-reacted components, tristearate, tristearate/distearate mixture and mono- or distearate, respectively (1, 3, 17, 18).

Total ash content (Table 1) is in agreement with the literature data (1, 3, 17) and indicates that aluminum stearates are mostly the mixtures of mono-, di- and tri- forms, in which aluminum distearate is the main constituent.

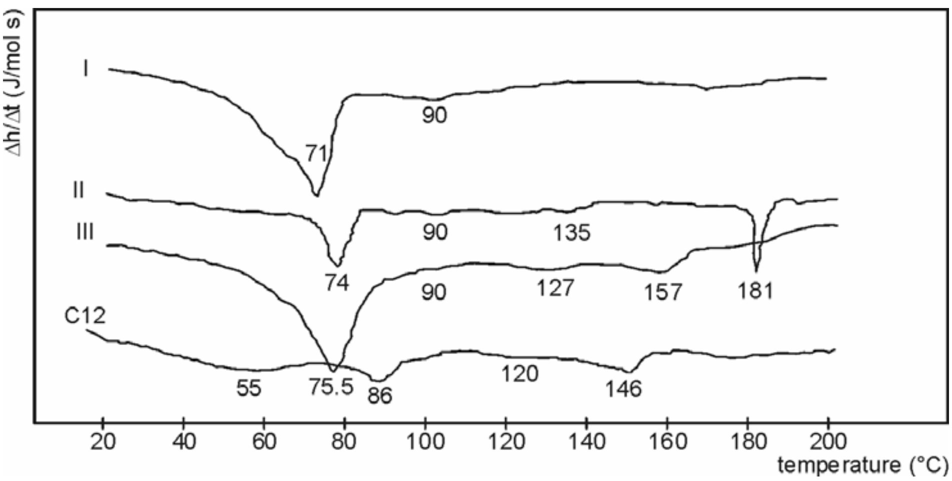

Fig. 1. DSC curves of aluminum stearate obtained for different molar ratios of stearic acid/sodium hydroxide (I 1:1.2; II 1:1.4; III 1:1.5) and commercial aluminum stearate (C.12)

Content of free fatty acids is a parameter which determines the application of aluminum stearate and shows efficiency of precipitation. It decreases with increasing of SA/ /NaOH ratio (Table 1). This means that the most effective ratio is 1:1.5. The same conclusion can be derived on the basis of mathematical comparison of the contents of reacted fatty acid and aluminum in the water-insoluble ash.

Density is a characteristic that is less important for technical application of aluminum soap and, irrespective of the salt form, it is 1.01 g/cm^3 (1-3). All the analyzed soaps had density less than 1 g/cm^3.

CONCLUSIONS

Aluminum stearate characteristics depend on the acid/alkali ratio used in the precipitation procedure. The decrease of this ratio results in lowering of the soap melting point. With increase of the acid/alkali ratio, aluminum content is increasing and non-reacted fatty acid content is decreasing.

After the comparison of the unreacted fatty acid content, melting point, water-insoluble ash, aluminum content and thermal effects, it can be concluded that soap obtained with acid/alkali ratio 1:1.5 is the most similar to the commercial aluminum stearate soap.

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ДОБИЈАЊЕ И КАРАКТЕРИЗАЦИЈА АЛУМИНИЈУМ СТЕАРАТА

Ева С. Лончар, Гизела А. Ломић, Радомир В. Малбаша и Љиљана А. Коларов

У раду је испитано добијање алуминијум стеарата поступком таложења при различитим условима сапонификације стеаринске киселине натријум-хидроксидом. Доказано је да је најповољнији моларни однос киселина/база 1:1.5 и да је добијени сапун веома сличан комерцијалном производу. Ендотермни ефекти одређени диференцијалном скенирајућом калориметријом, као и други параметри, показали су да су произведени сапуни смеше, где је дистеарат доминантан облик.

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