THE CHARACTERISATION OF Al₂TiO₅ COMPOUNDS OBTAINED FROM THE LIQUID PHASE (DAF)

Ligia Stoica¹, Carmen Gabriela Plapcianu², Mariana Irina Toașcan², Angela Lupu¹, Olimpia Micu¹, Gabriela Alexe²

1- University POLITEHNICA Bucharest, Faculty of Industrial Chemistry, Inorganic Chemistry Department, 1 Polizu, Bucharest 7000, ROMANIA
2- National Institute for Physics of Materials, 105 bis Atomistilor, Bucharest-Magurele, POB MG 7, 76900, ROMANIA

Abstract: Al₂TiO₅ precursors can be obtained using the liquid phase variant synthesis: precipitate flotation method - Dissolved Air Flotation (DAF). The precipitate flotation method allows the separation/recovery of metallic ions from diluted solutions by the interaction of the precipitate insoluble hydroxospecies with a tensioactive substance [1]. The isolated species formed as a result of this process are concentrated in the foam separated at the top level of the flotation column by bubbling an inert gas (air, N₂) into the solution.

The insoluble polymeric hydroxospecies isolated from diluted AlCl₃ and TiCl₄ chloride solutions 10⁻³ M after adding the coprecipitation and tensioactive agent were heated to form aluminum-titanates powder. The formation of the oxide after applying the thermal treatment to the Al₂TiO₅ precursors separated from the foam could be observed with the help of thermal analysis and X-ray diffraction. SEM investigations performed on the powder resulted in the oxide-precursors heating process revealed different size distribution depending on the tensioactive agent concentration. The samples having a higher tensioactive agent/metallic ion molar ratio present a larger size distribution (10μm).

Simple or mixed oxide precursors (Al₂TiO₅ type compounds) can be prepared using the precipitate flotation method.

INTRODUCTION

Aluminium titanate oxide precursors can be obtained from the liquid phase using the nonconventional method of Flotation, DAF (Dissolved Air Flotation) variant. The precipitate flotation method allows the separation/recovery of different metallic ions from diluted solutions by the interaction of the precipitate insoluble hydroxospecies with a tensioactive substance [1]. The species isolated in the flotation process are concentrated in the foam separated at the top level of the flotation column by bubbling an inert gas (air, N₂, Ar₂) into the solution.

Applying the nonconventional method of precipitate-flotation mixed oxides precursors can be obtained from diluted solutions in the range 10⁻³-10⁻¹ M, which are used to obtain oxides powders.

The paper presents the results obtained by studying the Al(III)-Ti(IV) coprecipitation from chlorides salts, followed by dissolved air flotation.

The insoluble polymeric hydroxospecies isolated from diluted chlorides provided by different industrial technologies could be the oxide precursors [2]. Applying a peculiar thermal treatment they lead to Al₂TiO₅ type compounds.

RESULTS AND DISCUSSION

Aluminium titanates precursors were prepared from diluted chloride solutions 10⁻³ M which were contacted for 20 minutes at room temperature. As precipitation agent we used a NH₃ 25% solution and the pH was adjusted to 7.7-7.5 after the tensioactive agent (laurylamine) was added in various molar ratio (0.01+1). The polymeric hydroxospecies [TiO(OH)₂(Al(OH)₃)₂H₂O] isolated in the foam separated at the top level of the flotation
column at optimal flotation conditions, were dried in air and then were characterized by chemical analysis and IR spectrometry. The IR spectra revealed the formation of coordinate species with laurylamine $[\text{TiO}_2(\text{OH})_6\text{Al}_2(\text{L-A})_2]$ which represent the oxide precursors.

Thermal analysis data showed water elimination up to 100°C and laurylamine decomposition up to 500°C. The endo-peaks from 550-600°C indicate the beginning of oxide formation. After 750°C no transition was registered.

The $\text{Al}_2\text{TiO}_5$ powders obtained by applying the thermal treatment were investigated using XRD spectroscopy and SEM.

The XRD spectra revealed the presence of aluminium titante from temperatures around 1000°C. As figure 1 shows there are some differences between the samples depending on the laurylamine content. Samples with higher laurylamine concentrations present a better formed $\text{Al}_2\text{TiO}_5$ (samples 25 and 24) which mean that polynuclear species are predominating in these cases.

![XRD spectra for samples heated at 1000°C](image)

**Figure 1** XRD spectra for samples heated at 1000°C

- $\text{x} - \text{Al}_2\text{TiO}_5$
- $\bullet \alpha \text{Al}_2\text{O}_3$
- $\ast \text{TiO}_2$

SEM investigations confirmed that particles are formed (200μm, 1μm-fig. size particle are predominating (10-100μm).

Therefore, using different particle size distribution for the mixed oxide powders, important characteristics such as bulk density and controlling the particle size distribution...
SEM investigations confirmed that at lower L.A:Ti molar ratio values (0.5) small size particles are formed (200μm, 1μm - figure 2) and at higher L.A:Ti molar ratio values (1) large size particle are predominating (10-100μm - figure 3).

Therefore, using different concentration of the tensioactive agent various size distribution for the mixed oxide powder can be achieved which could determine some important characteristics such as bulk porosimetry, density and aggregation tendency. By controlling the particle size distribution one can

Figure 2 SEM for sample 23 having L.A.Ti=0.5

Figure 3 SEM for sample 24 having L.A.Ti=1
CONCLUSIONS

The flotation technique can be used for mixed oxide precursors synthesis and useful compounds recovery from diluted solutions.

$A_2TiO_3$ powders could be prepared by heating the oxide precursors at temperatures over 1000°C.

The tensioactive agent (laurylamine) amount can determine the particle size distribution which could influence the further utilization of the oxide powder (insulator or support for UHF applications).

References