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Mechanochemical synthesis of lithium niobate

<u>S.Khalameida¹</u>, V.Sydorchuk¹, J.Skubiszewska-Zięba², R.Leboda², V.Zazhigalov¹

¹ Institute for Sorption and Problems of Endoecology of NAS of Ukraine, 13 Naumov Str., 03164, Kyiv, Ukraine; e-mail: <u>svkhal@ukr.net</u>

² Faculty of Chemistry, Maria Curie-Skłodowska University, Maria Curie-Sklodowska Sq.3, 20031 Lublin, Poland

Lithium niobate (LN) possessing perovskites structure is ones of the most promising electroceramic materials. The most widespread method of perovskites preparation is a solid state above 1000°C. Under these conditions, preparation of powders with high specific surface area is impossible. Therefore, the synthesis of LN in nano-dispersed state is important task. For this purpose, mechanochemical method may be promising.

Reaction mixtures (LiNO₃, Li_2CO_3 or LiOH H_2O)/Nb₂O₅) were subjected to milling in the planetary ball mill at 600-1000 rpm in water and air. The obtained powders were studied by means of XRD, DTA-TG, DSC, TEM, FTIR, Raman, UV–Vis spectroscopy, and low-temperature adsorption of nitrogen.

Direct mechanochemical synthesis of LN is realized during dry milling of Li_2CO_3 - Nb_2O_5 mixture at 1000 rpm for 1-2 h using ZrO_2 balls. As-milled LN possesses specific surface area about 18 m²/g, crystallite size 10 nm, and narrower band gap compared with solid state sample (3.89 against 4.35 eV). The latter can be associated with its defective structure which is confirmed by XRD, TEM and UV-Vis spectroscopy. It is shown in the increasing of absorption of irradiation with wavelength > 400 nm and the shift of absorption edge towards longer wavelength which corresponds to narrowing of band gap E_g . Additional calcinations promote improvement of crystal structure. As a result, E_g increases to 4.13 eV.

Prepared samples of LN exhibit mechanocatalytic activity in process of dyes degradation in aqueous medium.

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