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ILMIY AXBOROTNOMA

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STUDY OF ADSORPTION PROPERTIES OF Mg^{2+} MODIFIED Li - Mn SPINEL

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Abstract. In this work, the oxide phase of the spinel structure $Li_{1.33}Mn_{1.67}O_4$ 4 partially replaced manganese with Mg^{2+} ions, and the effect of lithium manganese oxides on adsorption properties was studied. $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ oxide with a spindle structure doped with Mg^{2+} ion was synthesized by sol-gel method and calcined at 600 °C for 5 hours. Experiments were conducted to study the effect of pH, gelation time, adsorbent dose, contact time and Li^+ concentration. The synthesized oxides of $Li_{1.33}Mg_xMn_{(1.67x)}O_4$ were described using X-ray radiation. The characteristics of $Li_{1.33}Mg_xMn_{(1.67x)}O_4$ were studied by X-ray diffraction (XRD).

Key words: Lithium, adsorption, Li^+, Mg^+, dopant, spinel.

Mg^{2+} bilan modifikatsiyalangan Li - Mn shpinelini adsorbsion xossalarini urganish

Annotatsiya. Ushbu ishda shpinel tuzilishli $Li_{1.33}Mn_{1.67}O_4$ oksidi fazasida marganesni Mg^{2+} ionlari bilan qisman almashtirildi va litiy marganes oksidlarining adsorbsion xususiyatlariga ta'siri o'rganildi. Mg^{2+} ioni bilan modifikatsiyalangan shpinel tuzilishli $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ oksidi zol – gel usuli bilan sintez qilindi va 600 °S da 5 soat kalsinatsiya qilindi. Tajribalar pH, gellanish vaqti, adsorbent dozasi, aloqa vaqti va Li^+ konsentratsiyasining ta'sirini o'rganish uchun o'tkazildi. $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ ning xarakteristikalari rentgen nurlari diffraksiyasi (XRD) bilan o'rganildi.

Kalit so'zlar: Litiy, adsorbsiya, Li^+, Mg^+, dopant, shpinel.

Исследование адсорбционных свойств Mg^{2+} модифицированной Li - Mn шпинель

Аннотация. В этой работе оксидная фаза структуры шпинели $Li_{1.33}Mn_{1.67}O_4$ частично заменила марганец ионами Mg^{2+}, и было изучено влияние оксидов марганца лития на адсорбционные свойства. Оксид $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ со структурой шпинделя, легированный ионом Mg^{2+}, был синтезирован золь-гель методом и прокаливался при 600 °C в течение 5 часов. Были проведены эксперименты по изучению влияния pH, времени гелеобразования, дозы адсорбента, времени контакта и концентрации Li^+. Характеристики $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ были изучены с помощью рентгеновской дифракции (XRD).

Ключевые слова: Литий, адсорбция, Li^+, Mg^+, допант, шпинел.

Introduction. Recently, the demand for lithium has been increasing for promising applications in many fields such as heat-resistant ceramics, welding flux, batteries, pharmaceuticals, etc. Lithium is extracted from mines and salt lakes, which currently contain a total of 17 million tons of lithium. Seawater is also a large source of lithium (about 2.5×10^{11} tons), but the lithium concentration is very low, 0.17 mg/l. Year by year, thanks to the development of modern technologies, lithium-ion batteries are widely used. Modern lithium-ion batteries and lithium-polymer batteries have the highest energy performance among rechargeable electrochemical systems.

Today, due to the above needs, the demand for lithium in the world market is rapidly increasing, and natural mineral lithium resources are significantly decreasing. However, the cost per unit of energy is still much higher than conventional systems with batteries. Cost reduction can be achieved by replacing the expensive cathode material with cheaper materials. For this, it is preferable to use Li-Mn-O oxide compounds with a spinel structure.

The advantages of Li - Mn systems include high specific capacity, acceptable electrical conductivity, and lack of toxic effects on the environment and people. Also, $LiMn_2O_4$ spinels show high potential. The sol-gel method is a convenient method for the synthesis of $LiMn_2O_4$ spinels. It is important to control optimal conditions for sol-gel synthesis: Li/Mn ratio, pH, concentration of metal salts, precursors, transition processes of hydrogel, etc. [1].

In traditional practice, precipitation is the most common technology for lithium recovery. However, it is difficult to apply the deposition method to high Mg/Li, marine and low-concentration lithium-ion battery waste solutions. Because Li_2CO_3 is insoluble (about 1.3 g/100 ml H_2O), it causes incomplete precipitation [2]. Therefore, the adsorption process is recognized as the most efficient method of lithium recovery from economic and environmental points of view [3].

However, a significant reduction of manganese was found in acid extraction of lithium during the adsorption process [4]. A very effective way to increase stability is to synthesize the spinel phase $LiMg_xMn_{2-x}O_4$ replaced by manganese by modifying it with divalent or trivalent ions (M = Mg, Co, Ni, Fe, Ti, Zn, Cr, etc.) [5-9]. From a practical point of view, the advantage of HMnO (Mg) is that one of the raw materials, $Mg^{2+,}$ is non-polluting and easily obtained from saline waters. Liyan Tian, Wei Ma et al synthesized magnesium(II)-doped spinel lithium manganese oxide (LMS) and prepared nano-free ionic sieve manganese oxide (HMS) by extracting lithium and magnesium from LMS. The processing of HMS explained that it can be used as a Li^+ adsorbent with topotactic extraction of lithium. proved that up to 99.2% Li^+ can be recovered from solution in 24 h under optimized batch conditions [10]. Priti Singh, Anjan Sil, et al. spinel powders of systems were synthesized by sol-gel technology using lithium acetate, manganese acetate, magnesium acetate, iron nitrate and citric acid [11].

The purpose of this work is to synthesize $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ ($0 \le x < 0.8$) spinels using the sol-gel method and study the effect of modification with Mg^{2+} ions on their textural properties.

Experimental. $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ spinel structure samples were synthesized by sol-gel method. For the synthesis, aqueous solutions of $LiNO_3$, $Mn(NO_3)_2$ and $Mg(NO_3)_2$ were used as precursors, which were mixed in a certain ratio to obtain oxides containing $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ ($0 \le x < 0.8$). Then a weighed portion of citric acid was added in the amount of 2 mol per 0.05 mol (Mn^{2+}+Mg^{2+}).

The resulting solution was evaporated at 80 °C until a thick gel was formed, and then dried at 100 °C for 8 h. The resulting powder was then calcined in air at 800 °C in a laboratory furnace (Fig. 1).

The characteristics of the samples were studied by X-ray diffraction (XRD). The adsorption and textural properties of adsorbents were studied using the nitrogen adsorption-desorption method in the ASAP 2020 porosity and surface analyzer.



a b

Figure 1. Appearance of the synthesized samples after drying at 100 °C (a) and after annealing at 800 °C (b).

Results and Discussion. X-ray diffraction lines of $Li_{1.33}MgxMn_{(1.67-x)}O_4$ sample are shown in Fig. 2. All of them show a pure spinel structure.

The diffraction lines of the delithiated material are almost identical to the previous diffraction lines, only the relative intensity is slightly weaker and the peaks are slightly shifted to higher values. This indicates that lithium extraction proceeds topotactically, maintaining the typical spinel structure.

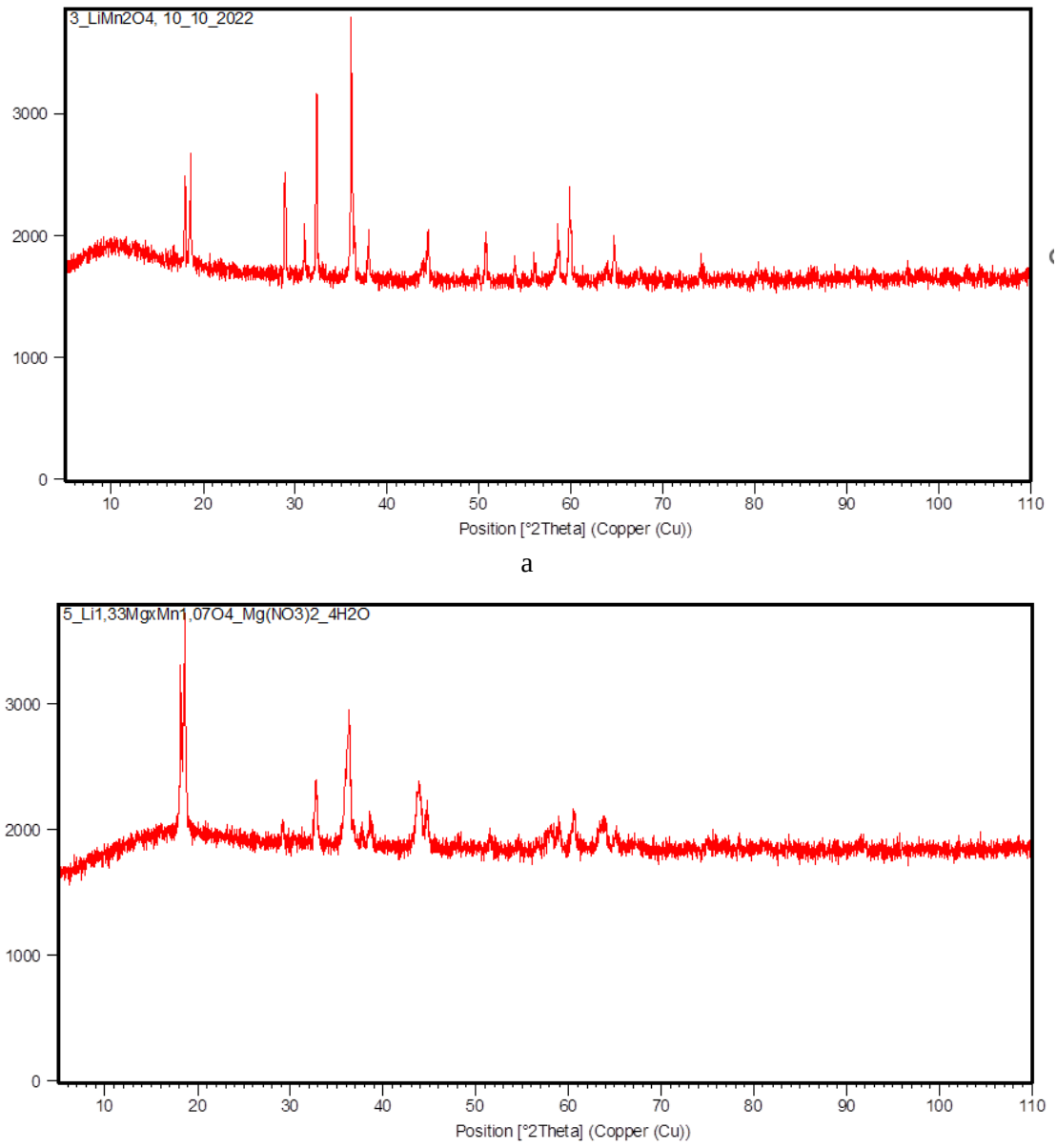


Figure 2. X-ray diffraction patterns of $LiMn_2O_4$ spinel (a) and $Li_{1.33}Mg_xMn_{(1.67-x)}O_4$ spinel (b).

Nitrogen adsorption-desorption isotherms of all samples have capillary-condensation hysteresis loops and belong to type IV according to the IUPAC classification (Fig.3. a,c, d), exhibiting characteristics characteristic of mesoporous bodies. The isotherms of the studied oxides are complicated by a hysteresis loop of the H3 type, which indicates the presence of cracks.

Samples of pure $Li_{1.33}Mn_{1.67}O_4$ (Fig. 3 b) and Mg-doped ($0.4 \leq x$) (Fig. 2 g, e) spinels have the same monomodal pore size with a maximum at $D \leq 3$ nm eat The $Li_{1.33}Mg_{0.8}Mn_{0.87}O_4$ sample (Fig. 3 z) is characterized by two bimodal distribution maxima at $D1 \leq 3$ nm and diffuse $D2 \sim 25$ nm, respectively.

The introduction of Mg^{2+} ions at $x \geq 0.4$ does not change the pore diameter values. The largest pore volume (0.073 cm^3/g) and average pore size (21.2 nm) are characteristic of the sample at $x = 0.8$, in which $Li_{0.5}Mg_{0.5}O_{1.5}$ and $Mn_{1.5}Mg_{0.5}O_{2.5}$ oxides there are mixtures. At the same time, in the range $0.1 \leq x \leq 0.4$, there is a 1.7-2.1 times increase in the specific surface area and 1.8 times in the pore volume.

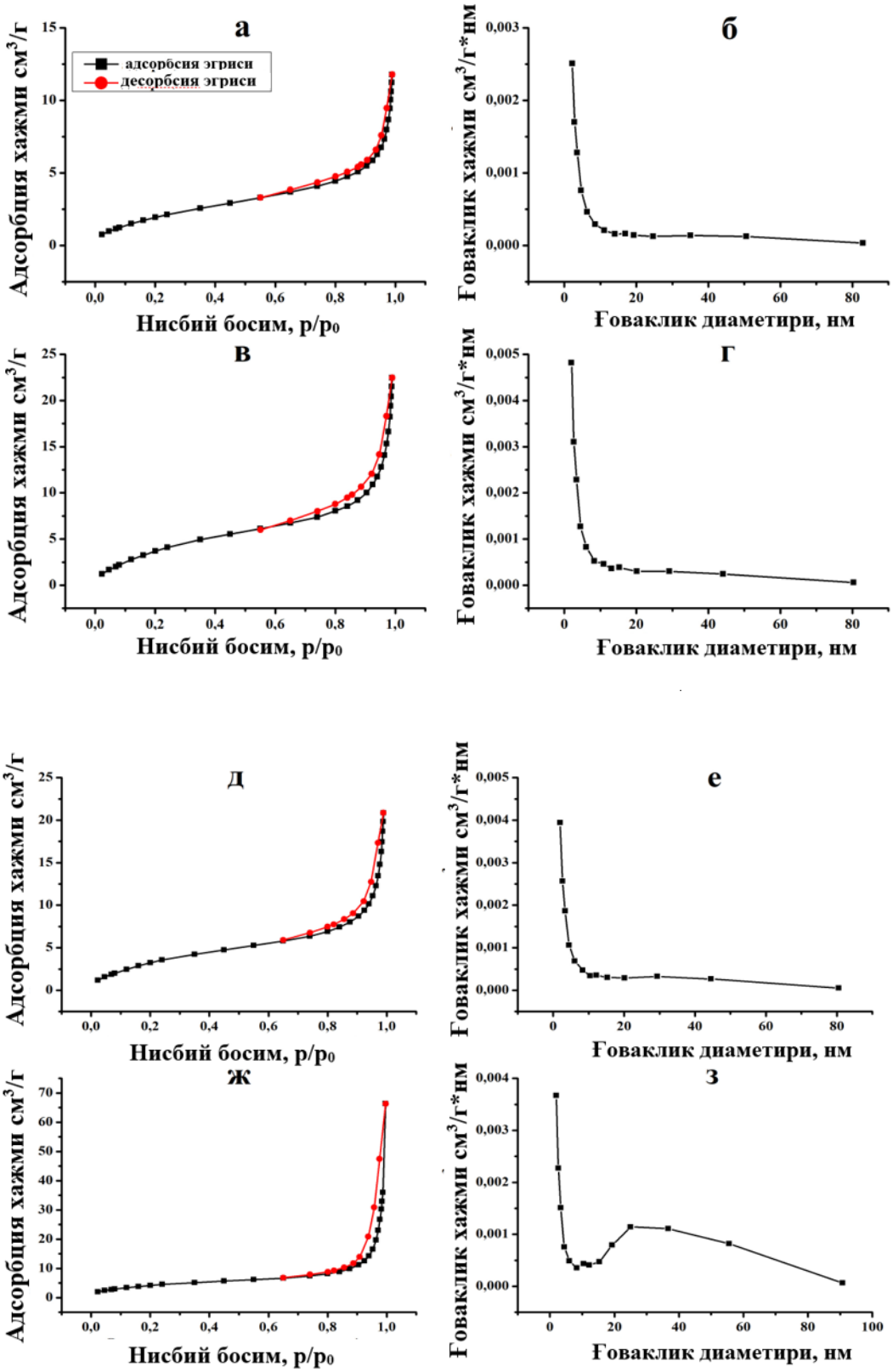


Figure 3. Low-temperature adsorption-desorption isotherms of N_2 (a, v, d) and BJH pore size distribution (b, g, e), samples $Li_{1.33}Mn_{1.67}O_4$ (a, b), $Li_{1.33}Mg_{0.1}Mn_{1.57}O_4$ (v, g), $Li_{1.33}Mg_{0.4}Mn_{1.27}O_4$ (d, ye), and $Li_{1.33}Mg_{0.8}Mn_{0.87}O_4$ (j, z).

An increase in the content of Mg^{2+} ions by x = 0.8 leads to a significant increase in the average pore size up to 21.2 nm (Table 1).

Table 1.

Textural characteristics of spinel samples

$Li_{1.33}Mg_xMn_{(1.67-x)}$ $(0 \leq x < 0)$

Sample	A_{BET}, m^2/g	V_{spdes}, cm^3/g	D_{BJHdes}, нм	Hysteresis loop type	Pore shape
$Li_{1.33}Mn_{1.67}O_4$	8,3	0,015	8,3	H3	cracked
$Li_{1.33}Mg_{0.1}Mn_{1.57}O_4$	17,1	0,028	7,9		
$Li_{1.33}Mg_{0.4}Mn_{1.27}O_4$	14,2	0,027	8,6		
$Li_{1.33}Mg_{0.8}Mn_{0.87}O_4$	16,6	0,073	21,2		

Conclusions. Thus, the study of the influence of Mg^{2+} ions on the texture properties of $Li_{1.33}Mg_xMn_{(1.67-x)}$ $(0 \leq x < 0.8)$ spinels showed the complex nature of the relationships obtained. Kinetic experiments showed that the adsorption process followed the pseudo-second-order model, and nitrogen adsorption of Li+ can be observed by the intraparticle diffusion model.

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