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STUDY OF THE ESTERIFICATION REACTION OF OLIGOPROPYLENE MACROMONOMER WITH SALICYLIC ACID

One of the widely spread technologies of preparation of the antimicrobial polymer materials is the addition of antibacterial additives to base polymers in preparation of polymer products process [1].

The washing of used low molecular additives from surface of prepared antibacterial composition materials (CM) for a short time deceases their exploitation time and influences negatively on their other properties. In order to extend exploitation time a large interest causes a use of the polymer additives containing biological active groups. The separation of polymer additives from composition materials gradually is practically impossible. In our previous investigations the information about representatives of oligomers containing salicylic group – oligoalkyl ethers of salicylic acid (SA) and acetyl salicylic acids (ASA), which can be used as polymer additives [2-3].

The purpose of this work is the development of technology of preparation of oligopropylene ethers of salicylic acid – new antibacterial oligomer additives containing salicylic group.

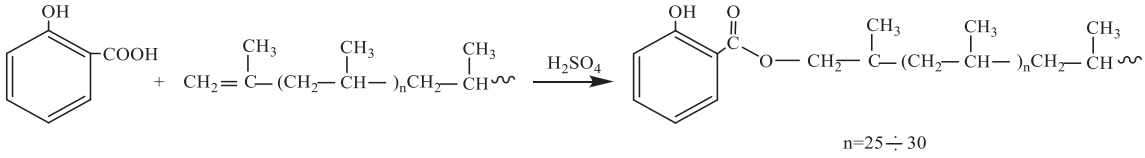
Oligopropylene macromonomer (OPMM) is obtained by a method of thermal decomposition in the special condition of isotactic polypropylene and its average molecular weight is 400-600, molecular weight distribution is close to 1.1.

The preparation of oligopropylene ether of SA. 10 gr of oligopropylene, 0.8 gr of SA and 0.1 gr of pyridine is dissolved in 30 ml of heptane. The mixture on water bath with reflux condenser is boiled at 70°C temperature for 1.5 h. After cooling of the reaction mixture the reaction product precipitates as a crystal. The product after being washed with ethanol is dried in vacuum. An average molecular weight is 630 (crioscopia), melting point – 148°C.

The IR-spectra of oligopropylene and oligopropylene ether of SA have been taken on "Agilent Cary 630 FTIR" spectrometry of firm "Agilent Technoloqies" in the range of 600-4000 sm^{-1}. For samples not forming qualitative thin layer their IR-spectra have been taken from transparent tablets prepared by pressing of mixture of these samples with this powdered ZnSe under vacuum.

Their molecular weight has been determined by a method of gel chromatography.

The purpose of this research work is determination of the peculiarities and optimal conditions of condensation reactions of salicylic acid with oligopropylene macromonomers. The reaction proceeds on the following scheme:



The composition and structure of the prepared oligopropylene ether of salicylic acid have been determined by methods of IR-spectroscopy (Fig. 1). The following absorption bands in the IR spectra indicate the presence of esters:

• valent (1654 cm^{-1}) vibrations of the C=O ester bond;

• valent (1202 and 3228 cm^{-1}) vibrations of the C–O and O–H bonds of phenol.

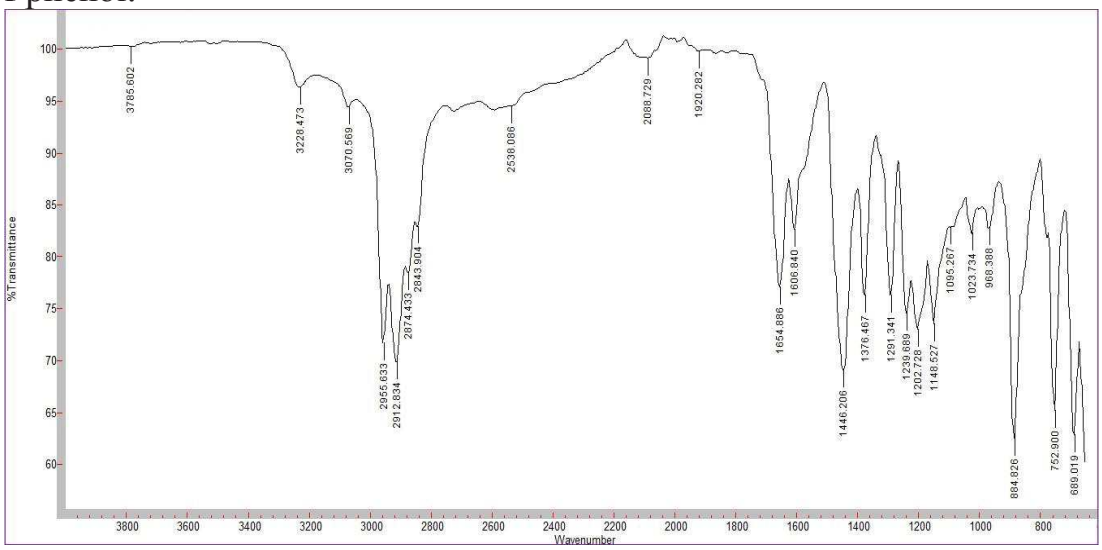


Figure 1 – IR-spectrum of oligopropylene ether of salicylic acid

The reaction has been carried out in solvents as DMFA, dioxane, toluene. The condensation reactions indices of salicylic acid with OPMM have been shown in Table 1 and Fig. 2.

Thus, the optimal reaction conditions can be considered as follows: temperature 60°C, solvent DMFA (80:20), reaction duration – 80 min.

As seen from Fig. 2 and Table 1 the highest yield is observed when DMFA is taken as a solvent. With increasing donor number and dielectric penetration of the solvent a yield of main product is also increased.

Table 1 – Influence of nature of the solvents on condensation reaction rate of salicylic acid with OPMM (T=60°C and a ratio of initial substances (SA-OPMM) 1:1)

Solvent	Yield, mol%	Reaction duration, τ, min.	Reaction rate, mol/l.min.10^2	Donor number (DN)	Dielectric penetration (ε)
DMFA	50.0	20	2.5	26.6	36.7
	64.0	40	1.6		
	71.0	60	1.18		
	75.1	80	0.94		
Dioxane	44.5	20	2.23	14.8	2.1
	51.6	40	1.29		
	60.8	60	1.01		
	64.0	80	0.8		
Toluene	32.0	20	1.6	2.4	1.4
	44.0	40	1.1		
	49.0	60	0.82		
	51.3	80	0.64		

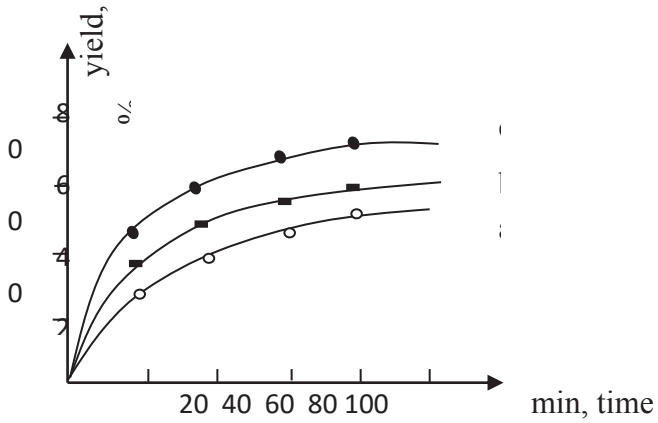


Figure 2 – Influence of nature of the solvents on condensation reaction rate of salicylic acid with OPMM: a-toluene, b-dioxane, c-DMFA

In general, in a case of use of the polar solvents due to electrophilic mechanism of addition the condensation reaction has the highest rate and yield. The molecular weight of oligoalkyl macromonomer and reaction temperature sharply influence on condensation reaction rate of this macromonomer with salicylic acid. The information about the results of investigations carried out in this field, and also about kinetic and thermodynamic indices of the reactions will be presented in the papers.

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ЭЛЕКТРОХИМИЧЕСКИЙ СЕНСОР НА ОСНОВЕ ПОЛИМЕРА С МОЛЕКУЛЯРНЫМИ ОТПЕЧАТКАМИ ДЛЯ ОПРЕДЕЛЕНИЯ ЛИНКОМИЦИНА

Устойчивость к противомикробным препаратам является глобальной проблемой здравоохранения, которая усугубляется чрезмерным использованием антибиотиков с потенциально серьезным воздействием на здоровье людей.

Для определения и распознавания лекарственных соединений, в том числе антибиотиков, все чаще используются электрохимические методы, в частности вольтамперометрия, которая успешно применяется с высокой селективностью и чувствительностью для анализа лекарственных препаратов и определения антибиотиков в фармацевтических лекарственных формах и биологических жидкостях.

За счет экспрессности, простоты пробоподготовки и дешевизны оборудования электрохимические сенсоры вызывают все больший интерес. Чаще всего при создании сенсоров для определения антибиотиков применяется модифицирование электродов полимерами с молекулярными отпечатками, или молекулярно импринтированные полимеры (МИП). МИП могут быть образованы с помощью различных методов полимеризации мономера вокруг молекулы темплата, а также может использоваться так называемый метод инверсии фаз, который характеризуется применением готовых полимеров, которые осаждаются на поверхность рабочего электрода из раствора в присутствии определяемого вещества.

Основной характеристикой МИП-сенсоров является селективность и чувствительность. Часто нанесение полимера на поверхность электрода приводит к уменьшению токов, поэтому необходимо добавлять допант к слою сенсора, который повышает чувствительность.