

SYNTHESIS AND PROPERTIES OF POLYMERS AND COPOLYMERS BASED ON N-CARBAMYLMALEIMIDE

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In the modern world, it is impossible to imagine human activities without polymer materials, which are found at every step in technology, health care and everyday life. Every day we come across various plastics, rubbers and synthetic fibers. Polymeric materials have various useful properties: they are highly resistant to aggressive environments; they are good dielectrics and heat insulators. Some polymers have high resistance to low temperatures, others – water-repellent properties.

In addition to an extensive list of advantages, high-molecular compounds also have disadvantages. Many polymers are prone to aging. For example, the process of reducing the chain length and size of the molecules is one of the types of polymer destruction. The process of chain reduction is due to the destruction of carbon bonds and the formation of radicals, which in turn contribute to the further destruction of polymer molecules. Destruction can be caused by mechanical loads, light, heat, water and especially oxygen and ozone.

It is known maleimide derivatives are an important building block in the field of organic synthesis, biochemistry and polymer chemistry [1]. Thermoset polyimides possess thermal stability, good chemical resistance, excellent mechanical properties and good dimensional stability even at high temperatures (300 °C and above). At the same time, they demonstrate high tensile strength. In addition, polyimides are used to produce composite materials reinforced with carbon or glass fibers [2].

The present work is devoted to synthesis of polymers and copolymers based on substituted maleimide, namely N-carbamylmaleimide, as well as the study of the physico-chemical properties of the compounds obtained.

Synthesis of N-carbamylmaleimide was carried out in two stages. In the first step N-carbamylmaleamic acid (maleuric acid) was obtained.

The reaction of maleic anhydride with urea was carried out in glacial acetic acid at 50 °C for 12 hours. The

mixture was allowed to cool and was left overnight at room temperature. The white crystalline product was filtered and washed with glacial acetic acid, and dried at room temperature. The resulting product was recrystallized from hot water.

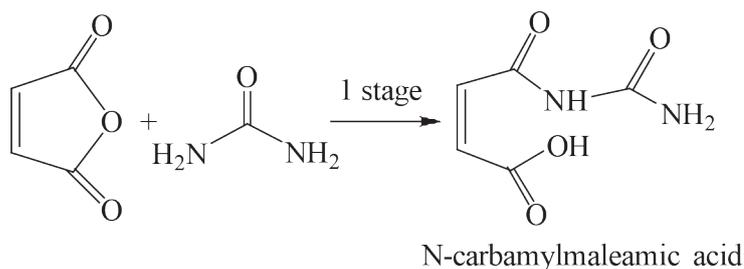
The yield of N-carbamylmaleamic acid 32–39% of theory. The melting point of the purified product is 158.6 °C, which corresponds to the data for N-carbamylmaleamic acid [3].

In the second step, N-carbamylmaleamic acid was cyclized by dehydration of water to form N-carbamylmaleimide.

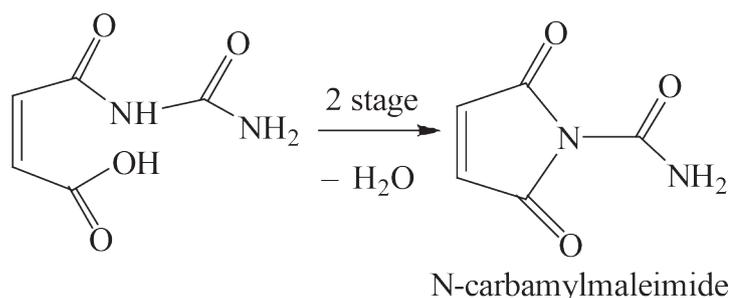
The synthesis was carried out at a temperature of 90–97 °C for 60 minutes. The yield of N-carbamyl maleimide was 30–35% of theory. The resulting product was identified using IR, ¹H-NMR and ¹³C-NMR - spectroscopy.

Radical polymerization of the obtained product was carried out in solution at a temperature of 80 °C for 7 hours and periodically (once per hour) sampled. As an initiator, azobisisobutyronitrile (AIBN) was used in an amount of 3% by weight of the monomer. After the polymerization was complete, the resulting polymer was precipitated in a 5-fold excess of hexane, filtered and dried.

Radical copolymerization is performed in solu-



Scheme 1.



Scheme 2.

tion at a temperature of 80 °C for 7 hours and periodically sampled. Styrene and butylmethacrylate were used as copolymers in an equal molar ratio with the starting monomer. As an initiator, AIBN was used

in an amount of 3 % by weight of the monomer. After the polymerization was completed, the resulting copolymers were precipitated in a 5-fold excess of hexane, filtered and dried.

References

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MODIFICATION OF NATURAL PETROLEUM ADSORBENT SPHAGNUM DILL

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Oil, consisting of a mixture of aromatic and aliphatic hydrocarbons, does not dissolve in water due to its hydrophobic properties, thus presents an environmental problem in case of emergency oil spills during oil transportation and refining because it degrades slower than water-soluble compounds.

The basic method to respond problems related to oil spills is sorption [1]. When choosing an adsorbent, it is necessary to consider not only its basic characteristics such as oil absorption, water absorption and buoyancy but also the methods of used adsorbent disposal. To respond oil spills, adsorbents shall be both oleophilic and hydrophobic. Synthetic fiber materials, such as polyethelene, polypropelene and atactic oxidized polypropelene, are good adsorbents of hydrocarbons. The adsorbents which combine liophilic and hydrophobic behavior are cellulose containing materials being able to participate in ion-exchange reactions [2]. The paper demonstrates that adsorption is determined by composition, structure and proportion of components. Natural adsorbents despite their diversity are characterized with high water absorption capacity and relatively low oil absorption capacity in comparison with polymer and inorganic adsorbents. The basic approach to increase adsorbent hydrophobicity is to modify cellulose containing adsorbents by treating them with chemicals: sodium hydroxide [3], Fenton's reagent [4], organic acids and acid amides [5], carbonization [6].

This paper studies target-specific modification of peat moss (*Sphagnum*) and the following three properties of the modified material: water absorption capacity, oil absorption capacity and buoyancy.

Experiment

Oil absorption (OA, g/g) and water absorption (WA, g/g): an oil slick of 0.5 to 5 mm thick was created over water surface in a drum with cross-section area of 250 cm. The sorbent was distributed over the oil slick. As the time passed, the sorbent was dewatered by filtering, and the adsorbed oil was extracted from the sorbent using carbon tetrachloride. Gravimetric method was used to determine the amount of the absorbed oil. Residual amount of oil in the water was determined with an IR spectroscopy method at the wavenumber of 2.926 cm⁻¹ and a thin-layer chromatography method.

A gravimetric method evaluating weight difference of the original adsorbent (M) and the water saturated adsorbent (M_1) was used to determine water absorption ($W\%$):

$$W = (M_1 - M) / M \cdot 100,$$

where Peat moss carbonization

A peat moss charge (4 g) was placed into a flask; the flask was closed to prevent the access of air into carbonization zone. The time of burning at the steady-state temperature from 100 to 400 °C was 60 minutes. Coalification degree of the peat moss was defined according to the formula: $R = C_\tau / C_0 \cdot 100\%$, where C_0 and C_τ are the weights of peat moss before and after carbonization process respectively. The difference between coalification degrees was determined as follows: $\Delta R = 100\% - R$.

The main criteria for using an adsorbent to clean water surfaces in nature are its water absorption capacity and buoyancy.