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V. L. Fleisher, PhD (Engineering), assistant professor (BSTU);
M. V. Andryukhova, PhD student (BSTU); D. S. Makarova, student (BSTU)

SYNTHESIS AND PROPERTIES OF INTERACTION PRODUCT OF ROSIN AND DIETHYLENETRIAMINE

Amides of rosin resin acids are a promising material for use in the technology of paper and cardboard in order to increase its strength characteristics. Therefore product of rosin resin acids interaction with diethylenetriamine arouses interest.

The influence of temperature and duration of the process of interaction of rosin resin acids with diethylenetriamine on the acid number of the product are examined in this article. Melting point of the received rosin amides was defined. The thermal stability of the reaction product of abietic acid with diethylenetriamine was studied. It was proved that this product contains the appropriate functional groups using IRspectroscopy.

Introduction. Nowadays many substances having amino or amide groups and capable of forming strong hydrogen bonds with the cellulose fibers are widely used to make paper and cardboard mechanically stronger. The ability of amines and amides to form hydrogen bonds allows their wide use as reinforcing additives in the production of paper and paperboard based on recycled cellulosic material. It is noted that the formation of hydrogen bonds between the amide and amine groups of reinforcing fiber materials and additives (particularly hydroxyl groups of cellulose macromolecules) improves the strength properties of paper and paperboard products. Nowadays polyamides are widely used as reinforcing additives in paper and paperboard production. The using of polyacrylamide allows significantly improve the strength of paper and paperboard [1]. It is probably can be based on the formation of numerous hydrogen bonds between the amide groups of polyacrylamide and cellulose hydroxyl groups. Polyamidoamines are as widely used as polyacrylamide. But they are used in the form of 35–50% aqueous

solutions to treat cellulosic materials to enhance their strength while wet [2]. Polymers or copolymers containing N-vinylformamide and acrylic acid are introduced into the pulp as the filler. It is also increases the mechanical properties of paper and paperboard [3].

Thus introduction of even small quantities of polyamides into sizing materials significantly increases the strength of the inter fiber bond, improves operational properties of the paper in dry and wet state, increases a breaking length and the degree of retention of fine fibers and filler particles. Therefore aminoamides of rosin resin acids may be promising materials for use in the technology of paper and paperboard in order to increase its strength characteristics.

Main part. The purpose of this work is study of mode parameters of producing of aminoamides in the process of interaction of rosin resin acids with diethylenetriamine (DETA).

The reaction of rosin resin acids with (DETA) can proceed with the formation of the products shown in Fig. 1.

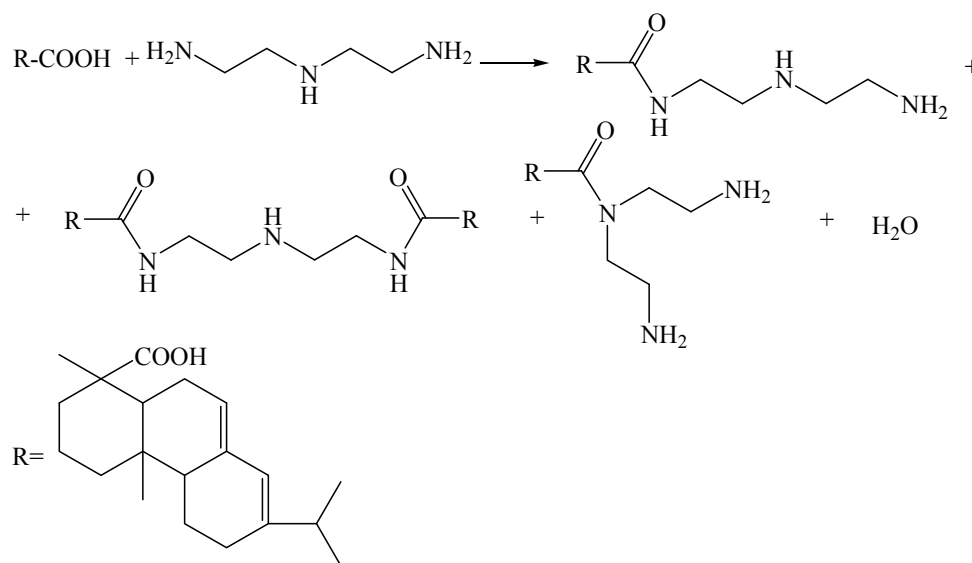


Fig. 1. Interaction products of abietic acid and diethylenetriamine

Pine Gum Rosin premium (JSC "Lesokhimik" Borisov) was used in the process of methodology developing. Study of the interaction of the resin acids of colophony with DETA was carried out in a heated reactor equipped with stirrer, Dean–Stark apparatus for distillation of evolved water and thermometer.

In order to obtain monosubstituted amide as the major product of the reaction the process is carried out at a molar ratio of 1.0 : 1.2 of DETA and rosin. The reaction was studied at temperatures of 190 and 210°C. There action path was controlled with the observing of the changes of the acid number of the reaction mass. Reduced acid number corresponds to a reduction in free resin acids in the reaction mass. The dependence of the acid number of the reaction duration is shown in Fig. 2.

Fig. 2 shows that carrying out of the reaction at a temperature of 190°C is impractical. In the process of the reaction the acid number had been down by only 2.5 times in 16 hrs. At a temperature of 210°C the reduction of acid number is more intensive and during first 4 hours of the process the acid number was down to 50 mg KOH / g which corresponds to the results obtained at 190°C in 16 hrs.

Obtained rosin aminoamides are dark yellow solid substance readily soluble in water without the precipitation formation which suggests the feasibility of further research in order to use them as part of reinforcing additives in the technology of paper and paperboard production. The melting temperature of the samples by the method [4] for

sample 1 obtained at 190°C is 24–26°C. For sample 2 this figure is 29–31°C. This difference is explained by the presence in the sample 1 of free DETA which contributes to noticeable reduction of the softening temperature.

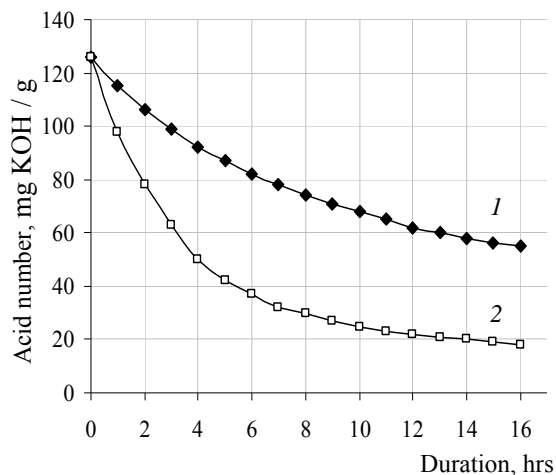


Fig. 2. Dependence of the acid number of reaction mass on the duration of the interaction of rosin with DETA at 190°C (1) and 210°C (2)

It was studied the thermal stability of the reaction product of abietic acid (as a main acid rosin) with DETA. These studies are of much interest because of some insufficiency in the literature of data about the thermal stability of the reaction product of rosin with DETA.

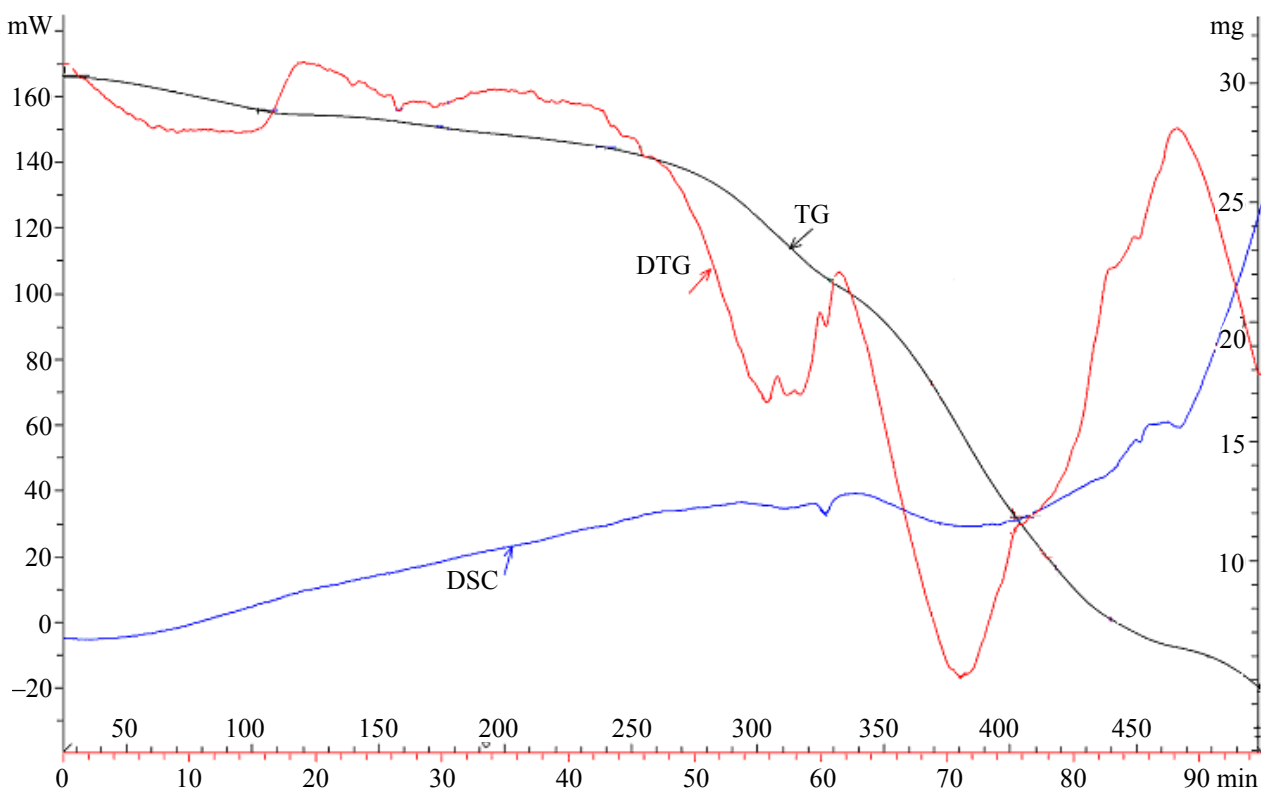


Fig. 3. The dynamics of thermal oxidative degradation of the reaction product of abietic acid with DETA

Finally it can have an impact on the properties of the end products, e. g. in reinforcing additives. Study of the dynamics of thermal oxidative degradation of the reaction product of abietic acid with DETA (Fig. 3) was performed by using the differential thermal analysis (DTA). Fig. 3 shows the thermogram has two obvious thermal effects with maxima at 306 and 383°C with mass loss of 28.2 and 85.3% respectively.

Thermal effect at 306°C is supposedly determined by the destruction of the amide group with the release of low-boiling substances and the thermal effect at 383°C by oxidative degradation of the conjugated system of abietic acid followed by the destruction of phenanthrene structure of the molecule.

IR spectroscopy was used to determine the presence of functional groups. IR spectra of test sample were recorded with using a spectrophotometer "FT-IR NEXUS" with Fourier transform in the frequency domain of 500–4000 cm^{-1} . The study sample was analyzed in the form of solid tablets pressed in potassium bromide.

In the IR spectrum of the sample prepared at 210°C intensive absorption bands are present at 1633, 1523 cm^{-1} which are in accordance oscillations of C = O in the secondary amide. The band at 3368 cm^{-1} indicates the presence of the group R-NH-R, i. e. the secondary amino group in DETA.

Conclusion. Thus in this paper we study the interaction of operating parameters of the reaction of rosin resin acids with DETA, in particular the

influence of temperature and duration of the process on the acid number of the product. The melting temperature of produced rosin amides was defined. The thermal stability of the reaction product of abietic acid with diethylenetriamine was studied. The using of IR spectroscopy proved the presence of appropriate functional groups in the product.

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