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AMIDE AND AMIDOESTER FATTY ACID DERIVATIVES AS MULTIFUNCTIONAL COMPONENTS OF PROTECTIVE ALKYD URETHANE COATINGS

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In order to expand the range of practically useful products based on renewable raw materials, a number of fatty acid derivatives, products of sunflower oil processing, were synthesized. The reaction of methyl esters of fatty acids with mono-, diethanolamine and piperazine yielded the corresponding amides of fatty acids. By reacting ethanolamide derivatives with maleic anhydride, maleated amidoester derivatives of fatty acids containing free carboxyl or hydroxyl groups were synthesized. A copper-containing product was prepared by the interaction of the dimaleinated derivative with copper acetate. All synthesized products showed solubility in alcohols and aromatic solvents. The obtained products were studied as multifunctional components of a film-forming system based on alkyd-urethane varnish brand AU(AL)-52W. It is shown that the synthesized ethanolamide and amidoester derivatives are regulators of the rheological properties of the varnish. Depending on the concentration, they can reduce (by 25-52%) or increase the dynamic viscosity of the varnish. Along with the effect on rheological properties, the synthesized additives in concentrations of up to 0.5-1.5% contribute to increasing the hardness of varnish coatings by 7.5-12.5% and do not negatively affect the drying time.

Keywords: alkyd-urethane varnish, amidoester, coating, ethanolamine, fatty acid, maleic anhydride, piperazine.

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Introduction

One of the primary objectives of modern polymer chemistry is to develop new polymeric materials using technologies that involve renewable raw materials instead of fossil hydrocarbons, which are irreversibly depleted. This approach not only eliminates the raw material problem but also helps to improve the environment and reduce greenhouse gas emissions [1]. Among the most promising renewable substitutes for fossil hydrocarbon raw materials, vegetable oils (VOs) are significant. Vegetable oils are widely used as a raw material for the production of alkyd resins [2,3]. Recently, there has been progress in research aimed at incorporating VO processing products into the production of polymeric materials [4,5], paint and varnish materials, mainly alkyd materials [6-8], and polyurethanes [9]. Ethanolamide derivatives of VO acids are of considerable interest due to their various applications. For instance, coconut fatty acid diethanolamide (CDEA) CAS 68603-42-9 is a widely used ingredient in many personal care products. Additives based on rapeseed and sunflower VO and diethanolamine are proposed for use in petroleum products [10].

One of the important representatives of the modern raw material base for the synthesis of binders (film-forming agents) and other ingredients for composite polymeric and paint and varnish materials is maleic anhydride (MA). In particular, a number of industrially important polymeric materials of polycondensation (unsaturated polyester) and polymerization types have been created using MA [11]. Bisphenols monoesterified with MA have been proposed as modifiers of some film-forming systems [12]. Structured polymers in the form of elastic film materials with ion-exchange properties were synthesized

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based on MA copolymers [13].

Recently, there has been an increasing interest in polyesteramides, which are polymers containing ester and amide groups in their macromolecular structure. These polymers are of great interest for use in tissue engineering and as delivery vehicles for dosage forms due to their combination of thermal and mechanical properties of polyamides with the biocompatibility and biodegradability of polyester [14,15].

Considering the aforementioned, research on the development of polymeric materials using amidoester derivatives from renewable plant materials, VOs, in conjunction with synthetic compounds such as ethanolamines and maleic anhydride, is highly pertinent.

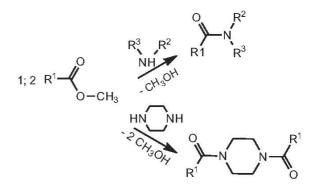
The objective of this work was to broaden the range of fatty acid derivatives that have practical applications. This was achieved by synthesising amides, ethanolamides, amidoesters, and carboxyamidoesters using VO and readily available synthetic raw materials such as mono- and diethanolamines, piperazine, and MA. The synthesised products were then evaluated for their potential as multifunctional components for modifying the properties of paints and varnishes.

Experimental

The starting substances, monoethanolamine, diethanolamine, piperazine, and MA, all of qualification «c» produced by LLC SPE Ukrorhsintez, refined sunflower oil produced by PJSC with II «DOEZ» (SO), alkyd-urethane varnish AU(AL)-52W produced by UNIPOL TU 2313-005-59846005-2007 (AU), were used without additional treatment.

A mixture of methyl esters of fatty acids (MFA) obtained by treating VO with an alkaline solution of methanol followed by separation from the glycerol fraction was used as a raw material for obtaining amide and amidoester derivatives of fatty acids (FA).

Amide derivatives of FA (AFA) were synthesized according to Scheme 1. For this purpose, appropriate amounts of MFA and amine and, if necessary, an alkaline catalyst were loaded into a three-necked reactor equipped with a stirrer, thermometer and Wurz nozzle with attached Liebich refrigerator, allonge and condensate collector. The reaction mass was heated with stirring. Condensation was observed at a temperature of 102–107°C. The condensate was heated and removed until the temperature of the reaction mass reached 137°C. By the amount of condensed methanol, the yield of AFA was 80–90%. Some characteristics of the obtained amides are given in Table 1.



Scheme 1. Synthesis of amide derivatives of FA (AFA). Here R1 is the hydrocarbon residues FA; R^2 is CH₂-CH₂-OH; and R³ is R², H

The resulting products were used without additional treatment.

Fatty acids (EAFA) were synthesized by condensation of maleic anhydride with AFA-1 and AFA-2 according to Scheme 2.

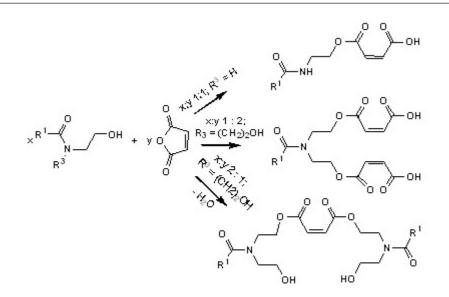
The calculated amounts of AFA-1 or AFA-2 and MA were loaded into a three-necked reactor

Table 1

Characteristics	Value or description			
code	AFA-1 AFA-2		AFA-3	
amine type	H ₂ N OH	HN OH	HNNH	
MEFA/amine ratio, mol/mol	1:1	1:1	2:1	
alkali content, mol/mol MEFA	0.015	0.013	_	
solubility	methanol, ethanol, xylene		aromatic hydrocarbons	
product appearance	waxy yellow plastic mass	red-brown transparent fluid mass	yellow-brown transparent fluid mass	

AFA characteristics

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Scheme 2. Synthesis of fatty acids. Here R1 and R3 are groups identical to those shown in the Scheme 1

equipped with a stirrer, thermometer and (in the case of the process according to Scheme 2 (c)) a Dean-Starke nozzle with a reflux condenser. The reaction mass was stirred without heating until the MA was completely dissolved. This was followed by an increase in the temperature of the mass from 18°C to 34°C.

In the case of the processes according to Scheme 2 (a and b), the mass was stirred at 45° C for 1 h and subsequently used without additional conditioning in the form of 50% solutions in xylene.

In the case of carrying out the process according to Scheme 2 (c), after dissolving the MA, sulfocationite KSM-2 produced by JSC «Smoly», dried to a constant mass, and xylene in the amount required to obtain the final product in the form of a 50% solution were added to the reaction mass. The mass was heated with stirring, gradually increasing the temperature to 120°C for 4 h and at 115–120°C another 1 h. The formation of water condensate during the stratification of the azeotrope in the Dean-Stark nozzle was observed.

Some characteristics of the obtained esteramide derivatives are given in Table 2.

To obtain a copper-containing product based on EAFA-2 (EAFA-Cu), 1.15 g of Cu(OAc)₂, 150 ml of ethanol were loaded into a three-necked reactor equipped with a stirrer, thermometer and reflux condenser and stirred at 45°C until the acetate dissolved. The mass was cooled to 30°C and a solution of 50 g of EAFA in 50 mL of ethanol was added. The contents of the reactor were stirred at 70°C for 1.5 h. After that, the reflux condenser was replaced with a Liebich condenser and the ethanol was distilled to a mass temperature of 115°C. The residue, a brown resinous mass, was dissolved in xylene to a concentration of 50%.

AEFA	charac	eteristics
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Table 2

Characteristics	Value or description			
EAFA code	EAFA-1	EAFA-2	EAFA-3	
AFA type	AFA-1	AFA-2	AFA-2	
AFA:MA (mol)	1:1	1:2	2:1	
solubility	methanol, ethanol, xylene			
appearance	resinous transparent masses of red- brown colour, slowly losing fluidity at			
appearance	room temperature			

Mixtures of AU with the synthesized products were prepared using a BGD 750/1 laboratory dissolver (Biuged Instruments, China).

The dynamic viscosity of the lacquer compositions at different concentrations of synthesised additives and shear rates was measured on a Brookfield viscometer MYR VI-062 manufactured by OHAUS at a temperature of 20^oC.

Samples for determining the hardness of the coatings were prepared by applying lacquer compositions to glass plates (60×90) mm in size using a Baker film applicator to obtain coatings with a thickness of 25 μ m.

The hardness of the coatings was determined using a pendulum tester type M-3.

Results and discussions

A feature of the structure of all synthesized products is the presence of both hydrophobic hydrocarbon residues of fatty acids (mainly oleic and linoleic) and polar amide groups in their molecular structure. In addition, AFA-1, AFA-2, and EAFA-3 products have polar hydroxyl groups, and EAFA-1 EAFA-2 EAFA-Cu products have carboxyl and unsaturated carbon-carbon groups of the monoesterified

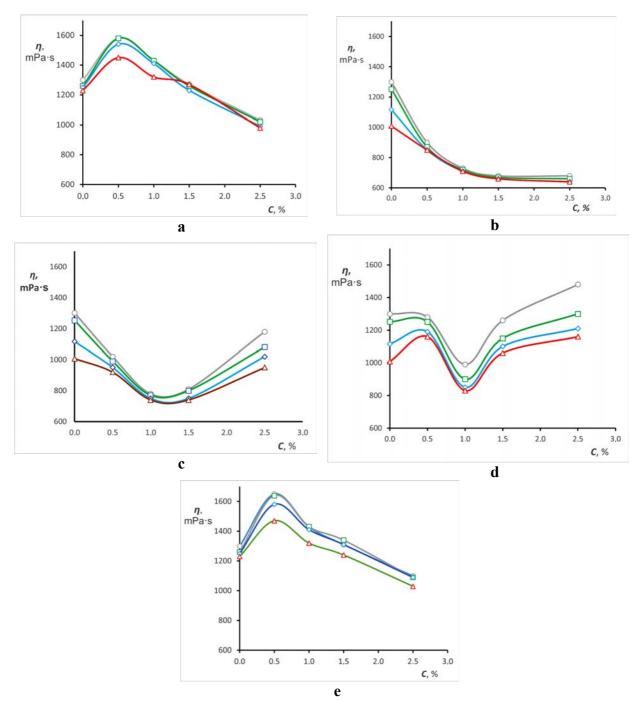
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maleic fragment. The presence of the above-mentioned functional groups provides grounds for predicting the presence of potential plasticizing, modifying, structuring and other properties of protective coatings in the synthesized products, depending on the nature of the main film-forming component.

Due to the non-technological consistency of the AFA-1 and EAFA-1 products, they were not used in

further studies. The remaining obtained products, AFA-2, AFA-3, EAFA-2, EAFA-3, and EAFA-Cu, were investigated as functional additives in the composition of alkyd-urethane varnish AU (AL)-52(W).

The results of rheological studies of UA at different concentrations of the synthesized additives are shown in Figure.



Dependence of the dynamic viscosity of AU on the content of synthesized products: a – AFA-2; b – AFA-3; c – EAFA-2; d – EAFA-Cu; e – EAFA-3. Shear rates (s⁻¹): o – 20; \Box – 50; \diamond – 100; and Δ – 200

Amide and amidoester fatty acid derivatives as multifunctional components of protective alkyd urethane coatings

According to the data obtained, the synthesized products have a significantly different effect on the rheological properties of AU, which is associated with the peculiarities of their chemical structure. Additives with free hydroxyl groups (AFA-2 and EAFA-3) at concentrations up to 1% increase the viscosity of the lacquer by 30-40%, depending on the shear rate. This is probably due to the interaction of hydroxyl groups with residual isocyanate groups of the lacquer base, which leads to its structuring. At a concentration of more than 1%, these additives exhibit plasticizing properties, contributing to a decrease in the dynamic viscosity of AU.

Amidoester derivatives (EAFA-2, EAFA-Cu) with free carboxyl groups at concentrations of 0.5-1.5% reduce the dynamic viscosity of the lacquer by 25-45%, depending on the shear rate. With a further increase in the content of additives in AU, an increase in viscosity is observed. The reason for this may be the formation of fluctuating mesh structures with nodes arising from hydrogen bonds.

In the case of the AFA-3 product, which does not contain free hydroxyl and carboxyl groups, a significant decrease (35-52%) in the dynamic viscosity of the lacquer is observed at the concentration range up to 1.5%. A further increase of the additive content, as well as the value of the shear rate, does not affect the viscosity of the system. This indicates the peculiarities of the plasticization mechanism caused by blocking the interactions between the macromolecules of the lacquer base.

Considering the drying time of the samples, the

most acceptable concentrations are as follows: up to 1.5% for EAFA-2, EAFA-Cu, and EAFA-3 products; and up to 0.5% for AFA-2 and AFA-3 (Table 3).

Tests of lacquer coatings showed that all synthesized additives, except for AFA-2, in the range of the studied concentrations, contribute to an increase in their hardness by 7.5-12.5% (Table 4).

The best effect of increasing the hardness of the coating is observed when using the additive EAFA-Cu, an amidoester derivative modified with copper ions. Probably due to the presence of copper, this compound also has the properties of a siccant.

Conclusions

The results of rheological tests of compositions based on alkyd-urethane varnish and synthesized compounds indicate that AFA-3, EAFA-2 and EAFA-3 can be considered as promising multifunctional modifiers of the properties of paints and varnishes. AFA-3 product at concentrations up to 0.25% reduces the dynamic viscosity and increases the hardness of the coating without affecting the drying time. Amidoester products EAFA-2 and EAFA-Cu have similar effects at concentrations up to 1.5%.

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Table 3

Additive content %	Drying time, hours, for samples with additives				
Additive content, %	AFA-2	AFA-3	EAFA-2	EAFA-Cu	EAFA-3
0	6	6	6	6	6
0.25	6	6	6	6	6
0.5	6	7	6	6	6
1.5	8	9	6	6	6
2.5	10	12	7	8	8

Drying time of AU samples with the addition of synthesized products

Table 4

Hardness values of AU coatings with the addition of synthesized products

Additive content, %	Hardness, M-3 conventional units, for coatings with additives				
	AFA-2	AFA-3	EAFA-2	EAFA-Cu	EAFA-3
0	0.40	0.40	0.40	0.40	0.40
0.25	0.40	0.42	0.40	0.45	0.42
0.5	0.40	0.43	0.43	0.45	0.42
1.5	0.38	0.40	0.43	0.43	0.45
2.5	0.38	0.35	0.40	0.39	0.45

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АМІДНІ ТА АМІДОЕСТЕРНІ ПОХІДНІ ЖИРНИХ КИСЛОТ ЯК БАГАТОФУНКЦІОНАЛЬНІ КОМПОНЕНТИ ЗАХИСНИХ АЛКІДНО-УРЕТАНОВИХ ПОКРИТТІВ

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3 метою розширення асортименту практично корисних продуктів на основі відновлюваної сировини синтезовано низку похідних жирних кислот – продуктів переробки соняшникової олії. Реакцією метилових естерів жирних кислот з моно-, діетаноламіном і піперазином одержано відповідні аміди жирних кислот. Реакцією етаноламідних похідних з малеїновим ангідридом синтезовано малеїновані амідоестерні похідні жирних кислот, що містять вільні карбоксильні або гідроксильні групи. Взаємодією діма- леїнованого похідного з ацетатом міді одержано мідьвмісний продукт. Усі синтезовані продукти виявили розчинність у спиртах та ароматичних розчинниках. Одержані продукти досліджено як багато-функціональні компоненти плівкоутворюючої системи на основі алкідноуретанового лаку марки АУ(AL)-52W. Показано, що синтезовані етаноламідні і амідоестерні похідні є регуляторами реологічних власти- востей лаку. Залежно від концентрації, вони здатні знижувати на 25-52% або підвищувати динамічну в'язкість лаку. Разом з впливом на реологічні властивості, синтезовані добавки в концентраціях до 0,5-1,5% сприяють підвищенню твердості лакових покриттів на 7,5-12,5% і не впливають негативно на час висихання.

Ключові слова: алкідно-уретановий лак, амідоестер, покриття, етаноламін, жирна кислота, малеїновий ангідрид, піперазин.

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Keywords: alkyd-urethane varnish; amidoester; coating; ethanolamine; fatty acid; maleic anhydride; piperazine.

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