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Urethane-Acrylates as Main Components of Lacquers for Protective Coating of Some Materials

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The synthesis of urethane-acrylic UV-cured resins intended for application as protective coatings for optical fibers. The utility of these compositions for protective soft, hardness and intermediate coatings of optical fibers was checked and confirmed under manufacturing conditions.

Keywords: UV-cured oligourethane-acrylates; protective coatings

INTRODUCTION

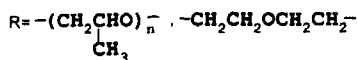
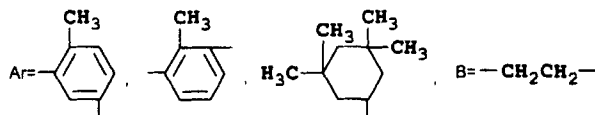
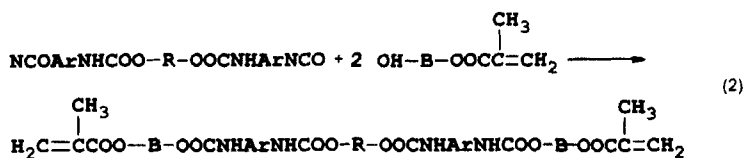
Oligomers containing the reactive acrylic groups at both ends are mainly used in industry. The materials cured by UV radiation are used as protective coatings for metals, plastic materials, wood, and optical fibers as the materials in electrotechnics and lithography¹⁻⁴.

During the last year our interest is concerned with synthesis and application oligourethane-acrylates as main components of the UV-cured compositions for the protective coatings of various materials and especially optical fibers⁵⁻⁷.

EXPERIMENTAL

In the first stage (Eq. (1)) a molar excess of isocyanate with the oligoetherols was introduced and a urethane prepolymer was obtained, containing some free isocyanate groups. Then, in addition reaction with hydroxyethyl- or

hydroxypropyl methacrylate (Eq. (2)) it produced the urethane-acrylic oligomer. Then the reaction was carried out until all NCO groups disappeared which was confirmed with IR analysis (no peak at 2274 cm^{-1}). As the materials there are used: the mixture of 2,4- and 2,6-toluylene diisocyanate (TDI) or isophorone diisocyanate (IPDI); glycols: oligooxypopylene M=2000 (PPG - 2000) and diethylene (DEG). A general scheme of preparation of the urethane-acrylic(UA) oligomers is represented by the chemical reactions.



Next stage of investigation was the research on the selection of the most suitable diluent. Active solvent decreases the viscosity as desired in accordance with the expectations and take part in the photopolymerization process of urethane-acrylate compositions. The following active diluents were applied: butyl (BA) and 2-ethylhexyl acrylates (EHA) and 1,4-butanediol diacrylate. This way, the composition urethane-acrylates was obtained. The chemical composition, the yield and the basic properties are presented in Tables I and II. Obtained oligourethane-acrylates compositions after adding Irgacure 651 as a photoinitiator formed materials curable with UV. There were exposed to the light of a Hg lamp (400 W, wavelength 320-380 nm) at a distance of 30 cm during 5 min. in non-oxygen atmosphere.

TABLE I Urethane-acrylic compositions

No UA comp.	Materials					Diluents	Contents of UA % weight
	Diisocyanates		Glycols		HEM		
	Kind	Weight [g]	Kind	Weight [g]	Weight [g]		
1.	TDI	174.2	PPG2000	976	130.1	EHA	85
2.	TDI	174.2	PPG2000	1175	105.0	BA-EHA	90
3.	TDI	174.2	PPG2000	976	130.1	BA	90
4.	TDI	174.2	PPG2000	285	104.1	BA	80
			DEG	47.8			
5.	TDI	174.2	PPG2000	285	104.1	EHA	75
			DEG	47.8			
6.	IPDI	222.3	PPG2000	285	104.1	BA	85
			DEG	47.8			
7.	IPDI	222.3	PPG2000	285	104.1	EHA	85
			DEG	47.8			
8.	TDI	174.2	PPG2000	240	130.1	BA	85
			DEG	40.5			
9.	TDI	174.2	PPG2000	240	130.1	BA-BDA	85
			DEG	40.5			
10.	TDI	174.2	PPG2000	240	130.1	BDA	83
			DEG	40.5			
11.	IPDI	222.3	PPG2000	240	130.1	BDA	85
			DEG	40.5			
12.	IPDI	222.3	PPG2000	240	130.1	BA	85
			DEG	40.5			

TABLE II Physicochemical properties of UA composition before curing

No UA comp.	Viscosity at 25°C [mPa*s]	n_D^{20}	d_4^{20} [g/cm ³]
1.	6000	1.4705	1.027
2.	9000	1.4698	1.026
3.	10500	1.4730	1.035
4.	17500	1.4868	1.081
5.	8700	1.4790	1.068
6.	14700	1.4801	1.079
7.	15500	1.4825	1.082
8.	12500	1.5098	1.102
9.	12700	1.5009	1.108
10.	10500	1.5147	1.110
11.	11500	1.5011	1.103
12.	13000	1.4989	1.099

TABLE III Mechanical properties of UA compositions after curing

No UA comp.	Properties					
	Gel content % weight	Shore's hardness A; *D	Young's modulus [M Pa]	Breaking stress [M Pa]	Relative elongation at break, %	Glass transition temp. [°C]
1.	93.2	96	2.0	0.87	70	-64
2.	91.0	52	2.8	1.01	65	-57
3.	88.5	75	4.5	1.56	50	-46
4.	92.0	75	47.0	9.40	40	-22
5.	93.5	60	15.0	5.20	35	-20
6.	95.5	68	69.8	8.80	45	-28
7.	98.0	80	75.0	10.10	56	-32
8.	98.8	46*	101.0	9.90	52	-7
9.	98.5	47*	80.2	9.90	42	-3
10.	99.0	58*	205.3	12.60	34	+4
11.	97.5	52*	227.4	11.20	45	-6
12.	97.0	50*	217.0	10.80	42	-11

CONCLUSIONS

The influence of the type and amount diisocyanates, glycols and active solvent on properties obtained oligourethane-acrylates was studied. Depending on the kind and quantity of the above materials we obtained such compositions which after curing meet the requirements for three different optical fiber covers. Some mechanical properties for three kinds of covers are as follows. Soft cover: Young's modulus – 2,0 MPa; hardness Shore's A – 52, water-vapor diffusion coefficient - $220 \mu\text{g}\cdot\text{mm}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$; intermediate cover: Young's modulus – 70 MPa, Shore's hardness A – 80, water-vapor diffusion coefficient - $75 \mu\text{g}\cdot\text{mm}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$; hard cover: Young's modulus 225 MPa, Shore's hardness D – 58, water-vapor diffusion coefficient - $12 \mu\text{g}\cdot\text{mm}\cdot\text{m}^{-2}\cdot\text{s}^{-1}$. Thermal properties of polyurethane-acrylates are presented in Table IV. The initial decomposition temperature and the temperature corresponding to the maximum decomposition rate are, respectively, 225-260°C and 330-370°C. This means that they show a relatively good thermal durability.

TABLE IV Thermal resistance polyurethane-acrylates

No UA comp.	Thermal analysis			Mass loss (%) at temp. (°C)			
	T_1^a °C	K_1^b % weight	T_2^c °C	250	300	350	400
1.	240	0.5	340	1.5	8.0	29.5	46.0
2.	225	1.0	340	2.0	8.5	31.5	56.0
3.	225	0.5	345	2.5	7.5	33.0	58.0
4.	225	0.4	345	2.4	10.0	30.0	58.0
5.	230	0.5	335	2.5	10.0	29.0	58.0
6.	225	0.6	335	2.5	9.5	35.0	58.0
7.	225	0.7	330	2.3	12.5	36.0	57.0
8.	240	1.0	370	1.5	8.0	29.0	55.0
9.	250	2.5	330	1.5	11.0	33.0	54.0
10.	260	1.0	360	0.8	6.0	22.0	49.0
11.	255	1.8	330	1.5	10.0	31.0	55.0
12.	225	4.0	340	6.0	15.0	40.0	63.0

^a – Temperature of initial decomposition from the DTG curve^b – Mass loss at temperature T_1 ^c – Temperature of maximum velocity of decomposition from the DTG curve

The applicability of the obtained compositions for protective soft, hard and intermediate coating of optical fibers was confirmed in the technological process of the production of telecommunication optical fibers carried out in the Laboratory of Optical Fibers Technology of the MCS University in Lublin, Poland. The UA compositions obtained were perfectly cured on the optical fiber which was drawn at a speed up to 100 m/min.

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