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Features of copolymerization of ethylene with acetylene on complex organometallic catalysts*)

Summary — Factors were studied that influence the kinetics of consumption of the comonomers in the copolymerization of ethylene with acetylene on vanadium- and titanium-containing complex catalysts, and the structure of the resulting products. The kinetic method showed the reactivity of acetylene to exceed in this process that of ethylene. The ethylene—acetylene copolymers (EAC) are colored; they contain conjugated and vinyl double bonds, react with ozone, bromine, iodine and maleic anhydride, are paramagnetic, and contain essential amounts of the crystalline phase. All these facts point out that they are block copolymers. Although nonelectroconductive, EAC are strongly electrified; they melt in the 80-140°C range, contain essential amounts of the crystalline phase both in the nascent state and in films: they are completely soluble in tetralin at 130°C and have low MFI values. Thermomechanical, calorimetric (DSC) and thermogravimetric data show EAC to be crosslinkable without initiators and accelerators. The acetylene blocks initiate crosslinking and operate both as a crosslinking agent and a fragment of the resulting structured material. The supermolecular structure, the structure and the strain—strength properties of EAC are similar to those of LDPE. Key words: copolymerization of ethylene with acetylene, organovanadium and titanium catalysts, ethylene-acetylene copolymers.

The interest in the copolymerization of ethylene with acetylene is evident in the searching for ways to introduce reactive double bonds into the basic chain macromolecules of polyethylene. Reception of such products would allow to decide questions about the crosslinks in the macromolecules and also to introduce other reactive groups into the copolymer.

The purpose of the present work is to study copolymerization of ethylene with acetylene on complex organometallic catalysts and the influence of various factors (nature and mole ratios of components in the catalyst, mole ratios of components in an initial mixture, pressure, temperature) on the kinetic differential consumption of the components and on the structure and properties of the resulting ethylene—acetylene copolymers (EAC).

EXPERIMENTAL

Copolymerization of ethylene with acetylene was studied in hexane as medium over the following catalytic systems: VOCl₃—Al(i-Bu)₃; TiCl₄—Al(i-Bu)₃; Ti(OBu)₄—Al(i-Bu)₃; VOCl₃—(C₂H₅)₂AlCl; TiCl₄—(C₂H₅)₂AlCl; Ti(OBu)₄— (C₂H₅)₂AlCl; VOCl₃—C₂H₅AlCl₂; Ti(OBu)₄— C₂H₅AlCl₂ and VOCl₃—Al(C₂H₅)₃ at temperatures of 273 to 333 K and pressures of the mixture of comonomers from 0.3 up to 1.0 MPa.

The composition of the gas phase sampled from the cylinder, and also from the reactor before the beginning and during the copolymerization was studied chromatographically by using an LHM-8-MD instrument equipped with a flame-ionization detector and with an Al203 A-1-on-apiezone L (5 wt. %) column.

The structure of EAC was studied by methods of ozonolysis, by EPR, IR and electronic spectroscopy, and also by the X-ray method. Specific heat, melting temperature, and crystallinity were determined on a DSM-2M differential scanning microcalorimeter.

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Thermogravimetry of EAC was studied in a MOM Q-1500D model derivatograph equipped with special quartz crucibles used in air and in an inert atmosphere.

MFI was examined by a standard technique in the IIRT-A device at 463 K and under a constant dead-weight load of 10 or 20 kg.

RESULTS AND DISCUSSION

Preliminary studies showed copolymerization of ethylene with acetylene to proceed most actively over the MtX_n — AlR_3 systems where AlR_3 was $Al(C_2H_5)_3$ or Al(i-Bu)₃. With the VOCl₃—Al(i-Bu)₃ system, variation of the Al/V mole ratio over a wide range (5—15) resulted in only insignificant changes of the integrated charge of comonomers. With the MtX_n — $(C_2H_5)_mAlCl_{3-m}$ systems, an increase in the mole ratio Al/MtX_n resulted in an essential increase in activity and productivity. A similar behavior was observed in the Ti(OBu)₄—C₂H₅AlCl₂ system. The nature of the organoaluminum compound was found to play an essential role in the formation of copolymerization active centers. Comparison of the activity and productivity of the catalytic systems showed that, at fixed parameters, preference should be given again to systems based on VOCl₃. Therefore, basic studies on the copolymerization of ethylene with acetylene were carried out on the catalytic system VOCl₃—Al(i--Bu)3 (1). Under the action of the system (1), acetylene was found to polymerize with the formation of black polymers insoluble in organic solvents. The rate of the reaction decreased quickly before polymerization was terminated (Fig. 1).

The decrease of the polymerization rate is caused by the instability of the catalytic system consequent upon the reduction processes resulting in the deactivation of

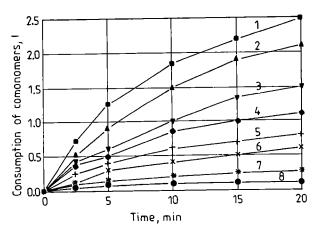


Fig. 1. The kinetics of consumption of comonomers in copolymerization run in heptane at 0° C over $VOCl_3$ — $Al(i-C_4H_9)_3$ in relation to the mole ratio in the mixture of comonomers: $VOCl_3$ —5.9 mmols/L; Al/V=1.5; C_2H_2/C_2H_4 : 1—0:1; 2—1:1500; 3—1:761; 4—1:345; 5—1:161; 6—1:62; 7—1:37; 8—1:0

active centers [1, 2]. In acetylene homopolymerization and copolymerization with ethylene, the deactivation rate of active centers increases presumably on account of interaction of the system (1) with the monomer and polyacetylene or EAC. Figure 1 shows that the activity, productivity and lifetime of the catalytic system (1) are higher in the copolymerization than those in homopolymerization of acetylene but lower than those in homopolymerization of ethylene. As the concentration of acetylene in the initial comonomers mixture and in the zone of the reaction is increased, the copolymerization rate and the productivity of the catalytic of system decrease and the copolymerizates turn lilac from white and further black-violet. However, even at 20-30% of acetylene in the initial mixture of comonomers, copolymerization proceeds at a significant speed and with good yield.

The decrease of the copolymerization rate with increase in the acetylene content in the initial comonomers mixture is probably caused by the formation of less reactive active centers in relation to ethylene as a result of introduction of the acetylene molecule into σ -Me-C species.

Another cause of the inhibiting action of acetylene may lie in its participation in chain breaking reactions as corroborated by a sharp decrease of the intrinsic viscosity of EAC (Fig. 2) and the appearance of vinyl double bonds in IR spectra of EAC (Fig. 3).

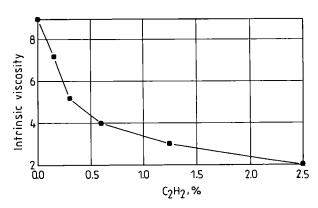


Fig. 2. Characteristic viscosity of EAC obtained over $VOCl_3$ — $Al(i-C_4H_9)$ in heptane at 0°C at total pressure of 0.3 MPa in relation to the content of acetylene in the initial mix with ethylene. $VOCl_3$ —5.9 mmols/L; Al/V=1.5

The mechanism of restriction of chain growth with the participation of acetylene is insufficiently clear. The fall of the molecular weight of the products at increased contents of acetylene in the comonomers mixture is a consequence of intrasphere resonant stabilization of active centers. Analysis of the gas phase at various intervals of copolymerization has shown the mole ratio C_2H_2/C_2H_4 to vary in the course of the process. Under static conditions of the copolymerization on system (1), in the absence or presence of fillers, acetylene was con-

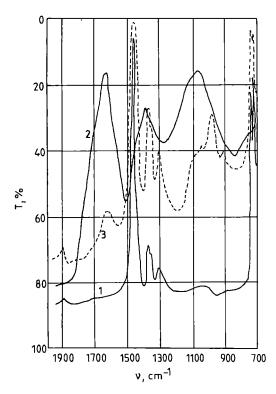


Fig. 3. IR spectra of polyethylene (1), polyacetylene (2) and EAC (3) prepared on VOCl₃—Al(i-Bu)₃

T a b l e 1. The influence of the acetylene contents in the initial mixture of comonomers and of the Al/V mole ratio in the system VOCl₃—Al(i-C₄H₉)₃ on the change of contents of acetylene in the gas phase in the reactor in acetylene copolymerization with ethylene in hexane at 288 K, total pressure 0.3 MPa. VOCl₃, 5.85 mmols/L

Al/V	t, min; C ₂ H ₂ ,%									
6.3	0 37.13	100 17.72	162 14.65	242 14.78	_					
6.2	0	4	22	66	90					
	3.42	2.98	2.98	2.28	2.53					
6.2	0	44	47	194	318					
	38.69	18.38	17.84	13.68	12.33					
4.7	0	4	31	77	111					
	10.45	4.41	4.02	4.02	3.94					
3.0	0	17	58	107	148					
	9.74	8.64	4.29	4.10	4.17					

sumed much faster than ethylene (Table 1), as evident from an essential decrease of its content in the gas phase in the reactor. Under quasistatic conditions, the gas phase actually shows a relative and an absolute increase in the ethylene contents. Copolymerization was carried out under conditions when the unique factor breaking the balance in the system was the submission of the mixture of comonomers; the difference in the solubilities of ethylene and acetylene in hexane played only a minor role in the estimation of the kinetic parameters of the process. The kinetic data observed, revealing changed composition of the gas phase in the reactor, qualitatively testified that the constant of the relative activity of acetylene is higher than that of ethylene.

The consumption rate of acetylene during the experiment depended both on the catalytic system used and on the copolymerization conditions. The initially high rate of decrease of acetylene concentration in the gas phase progressively diminished; a constant (quasistatic) concentration of acetylene was established in the experiments carried out over the VOCl₃—Al(i-Bu)₃ systems.

Theoretical analysis of the kinetic model of copolymerization under static conditions has shown that the mole composition of the copolymers formed on attainment of a quasistatic concentration of acetylene in the gas phase is equal to the mole composition submitted to the reactor with the initial comonomers mixture [1]. The contents of C₂H₂ in the products of copolymerization, received at the quasistatic concentration of comonomers, monotonously grows as the acetylene contents in the initial comonomers mixture is raised.

In some experiments with the VOCl₃—Al(i-Bu)₃ system and almost in each experiment with the titanium containing catalysts, the contents of acetylene in the gas phase of the reactor begins to grow over a certain interval of time after the quasistatic concentration of comonomers has been attained.

It is possible to explain this fact by the varying evolution of the catalysts used in the course of copolymerization. Deactivation of initial active centers in the system VOCl₃—Al(i-Bu)₃ results in the formation of products practically inactive in the polymerization. As opposed to this, in the TiCl₄—Al(i-Bu)₃ system, deactivation of initial active centers of polymerization on the Ti(IV) base leads to the occurrence of active centers of polymerization on the Ti(III) base [1].

The VOCl₃—Al(i-Bu)₃ is more active, but less stable than is the TiCl₄—Al(i-Bu)₃ system. The results obtained indicated that secondary active centers are less reactive with respect to acetylene. This is the reason of increase of the acetylene contents in the gas phase of reactor at the end of the experiment.

Coloring of EAC can be caused only by the presence of a system of conjugated chromophore double bonds in the macromolecule. In the electronic spectra of thin dark-violet EAC films there is a wide band close to 510 nm. Data [3] have testified to the presence of conjugated double bonds in violet EAC blocks including no less than ten acetylene moieties. The presence of conjugation of acetylene moieties in EAC proves to be true also of decolorized EAC upon interaction with maleic anhydride and tetracyanoethylene. IR spectral study of the adducts breaking the chain conjugated in acetylene blocks, occurs as a result of a Diels-Alder reaction.

Ozonolysis of ethylene-acetylene copolymerizates has testified to a significant nonsaturation. The bromine number of a EAC obtained on VOCl₃—Al(i-Bu)₃ (C_2H_2 —17%) was 9.25 g Br₂/100 g EAC. When doped with iodine, this EAC absorbed 11 wt. % of iodine.

EPR showed EAC to be paramagnetic. An EPR signal represents a single symmetric narrow (15e) line with a *g*-factor of 2.0028. The contents of paramagnetic particles

in copolymers changed within the limits of $1.4 \cdot 10^{16}$ up to $3.6 \cdot 10^{17}$ spin/gram and depended on the contents of acetylene in an initial comonomers mixture, and also on the nature of the catalyst components.

IR spectra of CEA (Fig. 3) over 690—1900 cm⁻¹ revealed bands of absorption characteristic for polyethylene chains, 1466, 1304, 632 and 721 cm⁻¹. The IR spectrum of polyacetylene showed bands at 1630, 1388, 1050, 1020, 699 and 670 cm⁻¹. The IR spectra of EAC contained bands of absorption at 1898, 1630, 1470, 1372, 1358, 1306, 1042, 995, 909, 730 and 722 cm⁻¹. Analysis of all groups of bands allows to obtain information on the structure of copolymers.

Take the change of the band at 720 cm⁻¹ on passing from polyethylene to CEA. This band is defined by rocking vibrations and fluctuations of regular polymethylene blocks, limited by moieties of other types. The specified blocks participate in rocking vibrations irrespective of the other part of the macromolecule, and therefore the intensity of the 720 cm⁻¹ band contains information about the average length of the polymethylene block.

With each characterized EAC, the ratio of the optical density of the 720 cm⁻¹ band to the optical density of the band due to methylene groups at 1460 cm⁻¹ is lower than that for polyethylene. The differences rise with increase in the contents of acetylene in the comonomers mixture. This fact shows that copolymers are formed, rather than homopolymers. The high absolute value of the specified ratio testifies to a large extent of polymethylene blocks.

The IR spectra of EAC, as well as of polyethylene, contain a well-expressed doublet at 720—730 cm⁻¹, indicative of a high degree of crystallinity of EAC. DSC and X-ray studies confirm this conclusion. The high crystallinity of EAC testifies that the ethylene blocks in EAC consist of no less than 15—20 monomer units.

The IR spectra of polyacetylene and EAC show a band of absorption at 1630 cm⁻¹, which is caused by valence vibrations of connected double bonds. The relative intensity of this band in the IR spectra of EAC is much lower than that in the IR spectra of polyacetylene. Similarity of IR spectra of EAC and polyacetylene indicates the presence of long blocks, consisting of acetylene parts in EAC; the presence of a band at 1020—1050 cm⁻¹ in IR-spectra of EAC is attributed to out-of-plane deformations of C-H in double bonds in trans-configurations of a high-conjugated polymer chain. The presence of conjugated acetylene moieties in EAC is also confirmed by decolorization of EAC on treatment with maleic anhydride and tetracyanoethylene.

Moreover, in IR spectra of EAC, there is a weak band at 670—699 cm⁻¹ attributed to out-of-plane deformations of C-H in double-bond *cis*-configurations.

The band at 1388 cm⁻¹ is defined by deformation vibrations of CH₃-groups. Weak bands at 910—995 cm⁻¹ testify about volume: some macromolecule parts of EAC contain at the end vinyl rather than methyl groups.

Comparison of IR spectra of polyethylene, polyacetylene and EAC shows the IR spectra of EAC to contain bands of absorption characteristic for polyacetylene and for polyethylene.

It was established that ethylene—phenylacetylene copolymerization on VOCl₃—Al(i-Bu)₃ in n-heptane at 0°C would yield colored copolymers, too.

Deep-colored copolymers are obtained even when in the reactionary environment for 100—150 ethylene molecules there is one acetylene molecule only. Formation of long acetylene blocks in the specified conditions is possible only in the event that the constant of the relative activity of acetylene has a much higher value than has the constant of the relative activity of ethylene.

At 130°C, EAC was completely soluble in tetralin and the solution was colorful. Polyacetylene was completely insoluble. This fact confirms that the products of joint polymerization are block copolymers.

An increase in the content of acetylene in the initial mixture of comonomers results in an increased content of acetylene in EAC and decreased intrinsic viscosity of EAC. A similar effect has been observed on copolymerization of phenylacetylene with propylene [1]. On VOCl₃—OAC and TiCl₄—OAC low–molecular EAC will be formed, described by rather high MFI (0.1—1.0 g/10 min). As opposed to titanium, on VOCl₃—OAC high–molecular EAC will be formed. At 190°C and 2 MPa, the MFI does not, as a rule, exceed 0.1 g/10 min [5].

The areas of application of polymers are in many respects defined by the temperature of transition from one physical condition into another. These transitions are well displayed on thermomechanical curves. Defor-

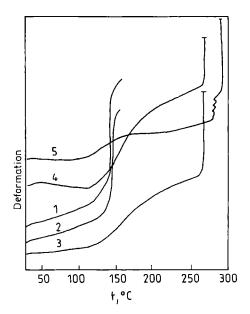


Fig. 4. Thermomechanical curves of EAC prepared over $VOCl_3$ — $Al(i-Bu)_3$ in relation to the acetylene content in the initial mixture of comonomers (Al/V=6.2; $T=15^{\circ}C$; P=0.3 MPa; curves 1, 3, 4, 5) and over $TiCl_4$ — $Al(i-C_4H_9)_3$ (Al/Ti=1; curves 2); acetylene,%: 1 — 0; 2 — 2.2; 3 — 8.4; 4 — 15.9; 5 — 23.3

mation tests of polyethylene and of EAC with small contents of acetylene show that, at 130—135°C, the material passes from a glassy condition at once into a viscous flow (Fig. 4). This is characteristic for thermoplastics which have crystal structures.

The increase in the contents of acetylene in EAC is accompanied by displacement of the temperature of transition into the viscous flow condition to lower temperatures. EAC containing 8-13% acetylene at temperatures exceeding 130°C, pass in the hyperplastic condition. EAC obtained from mixtures of comonomers containing more than 23% of acetylene, begin to soften and flow at 110—125°C, but on further increase of temperature the softening and the flow stop, and the material becomes firm and glassy (Fig. 4). Such thermomechanical curves unequivocally testify that, on melting, the high-unsaturated ethylene-acetylene block copolymers become thermo-crosslinked. A sharp increase of deformation of crosslinked EAC at temperatures exceeding 200°C, is associated with a specific destruction of the glassy material.

Crosslinking of EAC was investigated by the DSC and TG methods. Thermograms recorded in the heating mode at any content of acetylene in EAC usually contained one endotherm at 120—135°C and a more or less well-defined exotherm at 160—300°C (Fig. 5). The endothermal effect is due to the melting of ethylene blocks and the exotherm is the sum of thermal effects of isomerization of acetylene blocks, oxidation by air oxygen

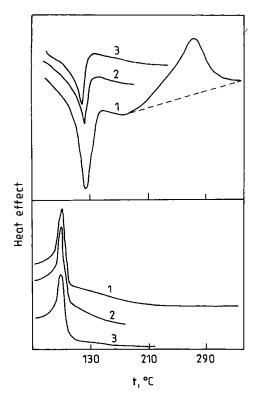


Fig. 5. Thermograms of the initial polymer containing acetylene and of the same copolymer in the subsequent reheating-recooling cycle: curve 1 — virgin run; curves 2, 3 — reheating-recooling runs

and thermo-crosslinking of the material. Isomerization of the *cis*-moieties in the *trans*-form in polyacetylene of Shirakawa occurred at 150°C; the present exotherm occurred at higher temperatures; the contribution of the heat of isomerization in the exotherm process may be neglected.

TG showed that, on heat treatment, EAC with the raised contents of acetylene grew by 1.5—3.3 wt % as compared with the initial weight. This indicates to an interaction of EAC with oxygen. Addition of oxygen to EAC on heat treatment in air occurs mainly due to double bonds and results in peroxide formation. Decomposition of peroxides can result in the formation of polymeric radicals. To preclude these processes, inert atmosphere was used in several cases.

Thermograms of EAC in the cooling mode contained only one exotherm at 85—100°C, caused by crystallization of ethylene blocks in EAC. In the repeated heating mode the EAC exotherm no longer appeared (Fig. 5). This fact indicates that, during the first heat treatment of EAC, all reactive groups were used.

Melting temperatures and crystallization of ethylene blocks are certain; the temperature of the begining, the maximum rate of cross-linking and entalpy melting, cross-links and crystallization of synthesized products can be evaluated from the thermograms. Some data are listed in Table 2.

T a b l e 2. The inluence of the acetylene contents in the initial mixture of comonomers, the ratio Al/V and frequency of heat treatment on the temperature and enthalpy of melting and crystallization of EAC over VOCl₃—Al(i-C₄H₉)₃ in hexane at 15°C; total pressure 0.3 MPa

C ₂ H ₂ %	Al/V	<i>T</i> ,,,,	<i>T_{j'}</i> °C			T	Enthalpy, J/g		
			start	max	end	<i>T_{cr}</i> ℃	melt- ing	joint	crys.
8.35	6.1	124	159	200	280	102	100.0	159.5	74.5
8.24	6.1	126	174	238	285	102	85.8	190.5	72.9
12.40	3.1	121	155	196	230	98	80.0	70.3	38.1
13.30	8.0	119	159	206	243	93	80.8	50.2	35.6
17.70	6.2	127	150	198	246	101	125.6	164.1	44.8
14.40	6.1	135	219	262	300	108	137.8	194.3	100.5

Formation of joints between macromolecules of EAC on heat treatment in air (and in inert atmosphere after contact with air) may be presented at the stages of initiation, growth and rupture of a chain. At the stage of initiation, active macroradicals are formed, capable of joining conjugated double bonds of EAC with the formation of new macroradicals. At the stage of growth, there occurs repeated recurrence of connection of macroradicals to acetylene blocks of EAC. Recombination or disproportionation of macroradicals lead to the rupture of the chain.

Thermocrosslinking of EAC represents thermal polymerization of acetylene blocks in the macromolecule of EAC. Assuming the enthalpy of this process to be close

to the enthalpy of ethylene polymerization (100 kJ/mol) and proceeding from enthalpy of thermo-crosslinking (167.5 J/g) and medium viscous molecular weight, it is found that EAC is containing 10—20 mol.% of acetylene cross-linked in several scores of tens cross bonds. The specific mechanism of EAC crosslinking is realized at temperatures exceeding 150°C, when EAC is in the molten condition. High temperatures provide activation and are transmitting mobility to macroradicals, necessary for their meeting and recombination. Crosslinking causes that the structure and properties of polymers usually strongly vary. Crosslinking influences the kinetics of crystallization, morphology and, what is particularly important, the deformation-strong property of a polymer.

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NOWOŚCI TECHNICZNE

c.d. ze str. 203

Firma DSM produkuje nowe typy poli(tereftalanu butylenu) Arnite. Są to materiały o znacznie zwiększonej płynności (co umożliwia wtryskiwanie wyrobów grubości zaledwie 0,4 mm, zmniejszenie ciśnienia wtrysku o 20% i zmniejszenie naprężeń własnych, dzięki czemu wyroby paczą się w mniejszym stopniu) i o krótszym czasie krzepnięcia w formie, dlatego też można na ogół skrócić cykl ich wtryskiwania o ok. 15%. Maja one zmniejszoną palność (VO w przypadku 0,8 mm) i zawierają 15% albo 30% włókna szklanego. Niektóre właściwości Arnite TV4 230 SNF z 15% włókna szklanego (w nawiasach odpowiednie właściwości Arnite TV2 260 SNF z 30% włókna szklanego): gestość — 1,51 (1,66) g/cm³, naprężenie zrywające — 100 (140) MPa, wydłużenie względne przy zerwaniu — 2,5 (2)%, moduł przy rozciąganiu – 6,5 (11) GPa, udarność wg Izoda z karbem w temp. 23°C— 6 (8) kJ/m², bez karbu w temp. 23° C — 30 (45) kJ/m²; HDT/1,8 MPa — 205 (210)°C; $HDT/0,45 MPa - 220 (220)^{\circ}C.$

Przewiduje się, że główną dziedziną wykorzystania obu typów będą cienkościenne elementy osprzętu elektrotechnicznego (łączniki przewodów, części wyłączników i silników, oprawy lamp, obudowy do kondensatorów itp.). Informacja prasowa firmy DMS z 5.08.1997.

Firma "Tosoh Corporation" z Japonii wprowadza na rynek **polimer o właściwościach adhezyjnych** *Melthene G*. Jest on produktem chemicznej modyfikacji kopolimeru etylen/octan winylu, przeznaczonym głównie do laminowania szyb. Przewiduje się także wykorzystanie tworzywa do wytwarzania szkła artystycznego.

European Chemical News 1997, 19—25.05., 27.

Fima "Cyro Industries" (USA) opracowała **płyty akrylowe o zmiennym efekcie wizualnym** *Acrylite GPP95*. Płyty te wydają się ciemne w przypadku oświetlenia światłem dziennym, natomiast stają się jasne w razie

podświetlenia własnym źródłem światła na ciemnym tle w nocy. Efekt taki uzyskuje się zmniejszając przepuszczalność światła płyty do 9% przez dodanie do przezroczystego materiału specjalnego pigmentu *Cyro color 1124-1* w ilości zależnej od żądanej przepuszczalności światła i od grubości płyty. Płyta ma gładką, błyszczącą powierzchnię od strony zewnętrznej, natomiast powierzchnia spodnia jest matowa, co powoduje zwiększenie rozpraszania światła. Płyty stosuje się do wykonywania dekoracyjnych szyldów, napisów itp.

Plastics Technology 1997, 43, nr 8, 25.

Firma "M. A. Hanna Color" (USA) oferuje przedmieszkę pigmentową *FX Forst*, która nadaje wyrobom z tworzyw przezroczystych modny efekt przeświecającego "mrożonego szkła". Może on występować w wersji bezbarwnej, barwionej oraz z dodatkowymi efektami satynowego matu lub iskrzenia.

Plastics Technology 1997, 43, nr 8, 46.

PRZETWÓRSTWO

Firma "Werner & Pfleiderer" (RFN) zmodyfikowała produkowane od dawna współbieżne wytłaczarki dwuślimakowe. Wersja MEGA Compounder ZSK ma w stosunku do dawniejszych modeli pogłębione kanały ślimaków, zwiększoną (nawet do 1200 obr./min) prędkość ślimaków i zwiększony o 30% moment obrotowy napędu. Umożliwia to zwiększenie wydajności 2—4-krotnie (w zależności od przetwarzanego materiału), a zwiększenie kosztu maszyny wynosi przy tym tylko 15%.

Informacje prasowe firmy Werner & Pfleiderer z 15.04. i 26.05.1997 r.

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