

O. M. Grytsenko¹, I. Gajdoš², T. O. Grytsenko¹, M. M. Yakovenko¹ Lviv Polytechnic National University, Department of Chemical Technology of Plastics Processing² Technical University in Košice, Department of Technologies, Materials and Computer Aided Production
oleksandr.m.grytsenko@lpnu.ua

TECHNOLOGICAL FEATURES OF THE FORMATION OF FILM PRODUCTS FROM MODIFIED HYDROGELS BY THE CENTRIFUGAL METHOD

<https://doi.org/10.23939/ctas2024.01.202>

Processing of polymers and composites based on them by centrifugal molding is important for production the bodies of revolution, which are used in various industries. This article analyzes the centrifugal molding and presents the results of research about new areas of such materials applications. For instance as method for the hydrogel films production of materials based on copolymers of 2-hydroxyethyl methacrylate (HEMA) with polyvinylpyrrolidone (PVP), including filled hydrogels, which can be applicable in medicine, electrical and tool engineering.

Key words: centrifugal molding; composite hydrogels; copolymers; polyvinylpyrrolidone; 2-hydroxyethylmethacrylate.

Introduction

Centrifugal molding is one method of plastic products production in the bodies of revolution by the filling of the material in a viscous-flow state into the form that rotates in the same plane [1–4]. Centrifugally material is evenly distributed on the inner surface of the form in a continuous layer, while receiving curing required configuration [5–7]. It is mainly used for the manufacture of pipes, bushings, bearings, gears and various pieces.

Centrifugal molding processing composition (compounds) based on polyester, epoxy and other connected, caprolactam, polyacrylates, low density polyethylene, polyamides and pasty molding compositions (plastisol) on the basis of polyvinyl chloride [8–13]. This method can also be recycled fine bulk polymers in the form of powders and granules [6, 14, 15]. Manufacture products from raw materials of the same type a monolayers products as well as multilayers, when using different types of materials (a mixture of polymers, oligomers or polymers that significantly different values of temperature fluidity). Curing compositions in the form under rotation occurs either through the curing reaction (for thermosetting materials), or because of a decrease in form temperature below the glass transition temperature T_c or the melting temperature T_m (for thermoplastics) [1–3].

Unlike centrifugal for rotational molding is a characteristic of form rotation around an axis – vertical, horizontal or inclined at an angle to the horizontal [7].

Analysis of the centrifugal molding process [5, 6, 13, 16] indicates the possibility of using this method for HEMA-PVP compositions processing in the presence of metal ions alternating oxidation and production of composite materials based on them. In addition, it contributes to high reactivity of these compositions [17, 18], the ability to regulate a wide range of initial compositions stay in the liquid state [19–21], the poor quality of the films obtained by other methods used at present – casting and filling into glass or polymer form. To produce films of polymer composite hydrogels by centrifugal molding is necessary to analyze the current state of technology at the moment and hardware design method and based on this new technology to implement the design, construction equipment and facilities.

The purpose of the research was to establish the possibility and develop technology of obtaining composite metal filled hydrogel films based on copolymers of PVP with HEMA (pHEMA-gr-PVP) by centrifugal molding. To achieve the purpose were set following objectives:

– design – and constructing of installation and equipment for centrifugal molding of hydrogel films;

– determination of process conditions of HEMA/PVP compositions processing.

Materials and research methods

Using the results of previous researches [17–21], for the synthesis of filled hydrogels selected polymerization in block composition of the HEMA: PVP = 80:20 mass parts in the presence of 0,05 %wt FeSO₄. The composition selected experimentally based on the velocity of polymerization of PVP content and FeSO₄, as well as the analysis of viscosity, based on the conditions of the filler particles sedimentation and the need for high performance process. For synthesis using 2-hydroxyethyl methacrylate ($\rho_{20}=1079 \text{ kg/m}^3$, $n_D^{20}=1.4520$), purified and distilled under vacuum (residual pressure $P_r=130 \text{ N/m}^2$, Boiling point $T_B=351 \text{ K}$), the content of residual ethylene dimethacrylates not more than 0.15 wt.%; PVP with MW 12 000 and 28 000 high purification before use dried under vacuum at 338 K for 2–3 h., fine powders of metals Zn, Co, Ni, Fe, Cu, Ag and FeCo alloy with particle size within 0.05–50 μm , the polymerization was carried out at 293 K, outdoors, in daylight.

Tensile strength at break (σ_{br} , MPa) and percentage elongation at break (ϵ_{br} , %) was determined by the method described in [22]. Water content (W , wt. %) investigated gravimetrically by the difference in weight and swelling coefficient (k) for the difference in size of the dry and swollen samples [17, 18]. Specific surface electrical resistance was determined according to the methodology [22].

Results and discussion

3.1. Construction of installation for centrifugal molding

Equipment for centrifugal molding is classified by technological and structural characteristics. There are specialized units for centrifugal processing compounds, plastisol and powder composite materials to construct different devices for dispensing and form (depending on the product configuration) [1–4]. Long tubes and sleeves are made in forms that rotate around a horizontal axis (Fig. 1, *a*). To produce products whose diameter is larger than the height, use forms that rotate around a vertical axis (Fig. 1, *b*).

Molds for centrifugal formation made of bronze, aluminum or steel. Since metals are preferred materials with high thermal conductivity.

As a prototype of the installation design for centrifugal molding was chosen mechanism of the centrifuge cylinder rotation, which is driven via a belt transmission (Fig. 1, *a*).

The current settings and molds for centrifugal formation are widely used for processing of polymeric materials, characterized by specific values of shrinkage due to which the formed product is easily removed from the mold. Materials that have low values of shrinkage, or characterized by high adhesion to the surface of the mold, such compositions based on epoxy and polyester resin, processed with the using of reference liquids (lead hexanate, mercury). pHEMA-gr-PVP copolymers also specify by high adhesion to the mold surface. However, adding of liquid adhesive coatings in this case is not possible because of the high reactivity of liquid HEMA/PVP compositions (possible interaction with salts as well as with mercury). In order to solve this problem is proposed the additional polymeric tube in the cylindrical mold made from material with low adhesion to HEMA/PVP composition [22]. This allows to smoothly removing the tube with work piece of the mold, followed by removal of the finished product, which excludes the possibility of destruction or damage, and increases the quality of the film surface.

Constructed installation (Fig. 2) contains a mold as hollow cylinder 1 which is driven through a belt transmission 13 by the electric motor 11. In the cylindrical body of mold 1 set polymeric tube 4, this is the pipe cut along the length. Mold is equipped on both sides of end cups 2 and 3.

End cups have ports for their connections with the transmission shaft 7 and hollow sleeve 8, made with a through axial hole for loading the composition and the possibility of creating a vacuum in the mold and, if necessary, fill it with an inert gas. The transmission shaft 7 connected by driving belt 13 with the electric motor 11. On the shafts 7 and 8 are mounted bearing parts 12 which are placed in the appropriate slot supports 5 and 6. To support 6 are able to regulate its height, and therefore provide horizontal arrangement of axis mold.

The process of centrifugal molding is relatively simple, which makes it possible to actively influence its course and reach the receiving quality products, competitive price and consumer properties. The process can be divided into the following stages:

- Preparing the mold.
- Preparation of raw materials (possibly mixing, separation, drying, dissolution, etc.). Mixing of

composition can occur directly in a mold that rotates, or outside the mold, followed by casting.

– Loading raw material in mold. Occurs through feeders or metering devices. During the products manufacturing loaded of reinforced compo-

sition may precede laying and compaction of reinforcing material.

- Rotations drive start-up.
- Product formation.
- Removing the product.

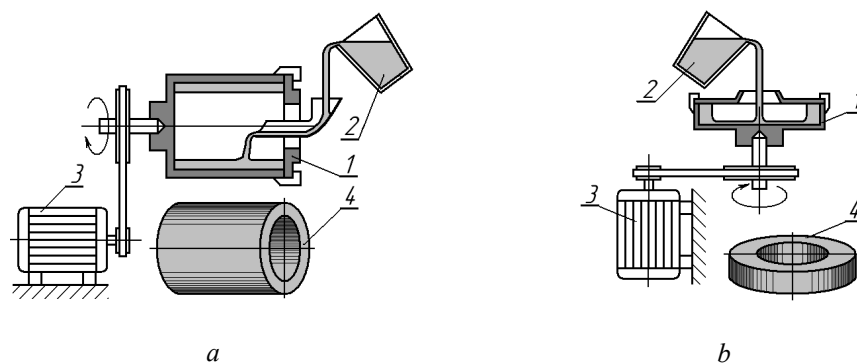


Fig. 1. Scheme of plastic centrifugal molding: a – around a horizontal axis; b – around a vertical axis: 1 – mold; 2 – polymer composition; 3 – electric motor; 4 – final article

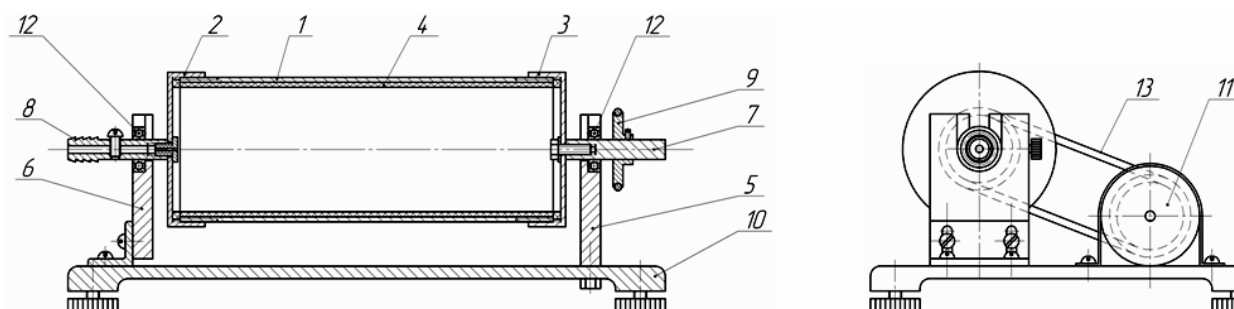


Fig. 2. Installation for centrifugal molding of composite films of polymer hydrogels: 1 – mold; 2, 3 – end caps; 4 – polymeric tube; 5, 6 – supports; 7 – transmission shaft; 8 – hollow sleeve; 9 – sheave; 10 – foundation; 11 – electric motor; 12 – bearing part; 13 – driving belt

Depending on the nature of the compositions during their processing, there are certain features of the products formation. Before starting you should establish the level of the mold and installation to the horizontal plane. The components of polymer composition are mixing outside and then load directly to the mold. Connect a hose to load the composition to the shaft 7 and fill in the mold 1. Disconnect a hose and turn on the electric motor 11. Engine speed is controlled by a universal speed controller. Depending on the physical, mechanical and rheological characteristics of the polymer composition and thickness of the product choose the rotation regime. For viscous compositions used regimes with high speed to intensively removing impurities and gas bubbles.

After the required time of molding, which depends on the degree of polymerization or curing of composition, switch off the motor 11 and take off driving belt 13 from the sheave 9. Remove mold from supports 5 and 6 and unscrew the end cups 2 and 3. Then push of polymeric tube 4 with work piece of the cylindrical body 1. Due to the elasticity of the tube, remove finished tubular product. If necessary, the product is cut along the length. Then the cycle is repeated.

According to the viscosity and nature of the composition there are couples of possible ways loading the composition into the mold 1 and working of the installation:

1. In the case of highly reactive liquid compositions that do not require evacuation, mold 1 filled by the polymer composition through the port of the shaft. Then plug in electric motor 11.

2. Viscous compositions loaded in the removed form 1 installed vertically. To do this, remove the driving belt 13 from the sheave 9, take off mold 1, unscrew the cup 2, load composition in the mold cavity, collect and set form in reverse order.

3. Compositions that require evacuation and run the polymerization in inert gas load as described in 1 or 2 cases. To shaft sleeve 8 connect a hose through which creates a vacuum and injects an inert gas. Then disconnect a hose and turn on the electric motor 11.

4. To obtain the reinforced films before assembly installation, you should enclose reinforcing material in polymeric tube 4. Place the tube into the mold, fix the mold and meter the composition one of these methods.

3.2. Technological parameters of processing

Process conditions of the centrifugal molding method include a rotational frequency of mold (n) and molding pressure (P_m), molding temperature (T_f), duration of molding (t_f), temperature and cooling time (for thermoplastics) [1–4]. For the molding of films based on pHEMA-gr-PVP we add another technological parameter – life time of the composition (τ).

Molding temperature

Logically, with increasing the molding temperature the rate of polymerization and, subsequently, the rate of film formation will elevate. However, HEMA-PVP composition in the presence of metal ions alternating oxidation [17, 18], and filled with metal powders [23], are characterized by high rates of polymerization are at room temperature in air, which is the main advantage of such materials. At the same time, implementation of mold with thermostatting system, complicates its design. If necessary installation can be set in the heat chamber or carry out infrared heating of the mold surface. So for work let's take the temperature close to ambient (20÷25 °C).

Duration of molding

Duration of molding is the time limited by the polymerization rate of the composition. Depend on the temperature, composition, initiator concentration, and for the filled compositions – the nature and

content of the filler. In particular, t_m will effect on the duration of the whole process of film formation. Established [19] that for the polymerization in solvent (water) the highest rate observed at $[\text{FeSO}_4] = 0.01$ wt. %, while polymerization in bulk – at concentrations of $\text{FeSO}_4 = 0.1$ wt. %. Therefore, in further researches used these concentrations of FeSO_4 . Duration of molding was taken as the time when the degree of conversion is 98–99 %.

Rotational frequency of mold, centrifugal force and pressure acting on the outer surface of the film

The frequency of mold rotation depends on the quality, physical and mechanical properties of the film. Rotational frequency affects the magnitude of the centrifugal force, and accordingly, the molding pressure and density of the film. The work [24] establishes a functional relationship that allows you to determine these parameters and the relationship between them.

Rotational frequency of mold n must exceed a certain critical value n_{cr} , wherein the material is held by centrifugal forces on the entire inner surface of the mold. If $n < n_{cr}$ the liquid material flows down from the walls of mold and the process of formation is not possible. Theoretical definition n_{cr} is significantly constrained and in practice n_{cr} found empirically. In order to form high-quality surface films the working rotational frequency of mold n_w must be greater than critical:

$$n_w > n_{cr} \quad (1)$$

$$n_{cr} > \sqrt{\frac{g}{4\pi^2 R}} \quad (2)$$

where R – radius of the inner mold surface (outer radius of the product), m (Fig. 3).

Of course, the quality of the film surface will primarily depend on the pressing force of forming film to the surface. The pressure will cause the centrifugal force on the composition layer is equal to:

$$P = \frac{F_c}{S} = \frac{F_c}{2\pi Rl} \quad (3)$$

where F_c – centrifugal force; $S = 2\pi Rl$ – surface area of the composition layer, m^2 ; l – work length of designed mold, m.

Mass of the elementary layer dm can be calculated as

$$dm = \rho \cdot 2\pi \cdot r \cdot l \cdot dr, \quad (4)$$

where ρ – density of the composition, kg/m^3 ; r – radius of the elementary layer of the composition, m; dr – the thickness of the elementary layer of composition, m.

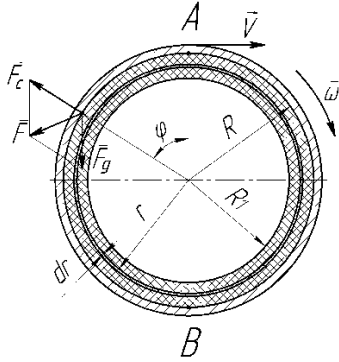


Fig. 3. Cross-section of a cylindrical mold with a composition and location of existing forces during centrifugal molding

Then, the magnitude of the centrifugal force acting on an elementary layer of radius r and thickness dr (Fig. 3) can be represented as:

$$dF_c = \frac{\rho \cdot 2\pi \cdot r \cdot l \cdot dr \cdot \omega^2 \cdot r^2}{r} = 2\pi \cdot l \cdot \rho \cdot \omega^2 \cdot r^2 \cdot dr, \quad (5)$$

Integrating equation (5) from R_1 to R , we obtain the equation for determining the centrifugal force acting on the composition:

$$F_c = 2\pi l \rho \omega^2 \int_{R_1}^R r^2 dr = \frac{2\pi l \rho \omega^2 (R^3 - R_1^3)}{3}, \quad (6)$$

where R – outer diameter of the film, m; R_1 – inner diameter of the film, m.

From equation (3) using (6) we determine the pressure acting on the outer surface of the film:

$$P = \frac{\rho \omega^2 (R^3 - R_1^3)}{3R} \quad (7)$$

$$P = \frac{4\rho\pi^2 n^2 (R^3 - R_1^3)}{3R},$$

or

Life time

Preparation and loading stages of composition are accomplished when the composition is in a liquid state. Life time is the time during which the composition is in a viscous-flow state and it limits the mixing and filling molds times. This is one of the key parameters during the formation of products from hydrogel materials. As the forming time it depends on the temperature, composition, concentration of initiator, the content and nature of the filler. In this paper, life time established by experimental method and depending on the composition, solvent and filler content was in the range 2÷60 min. As fillers used fine powders of metals Fe, Co, Ni, Cu, Zn ($D = 10\text{--}50 \mu\text{m}$). The shortest life time characterized for compositions containing zinc (Fig. 4, a) and the longest for copper (practically does not polymerize). Water dilution of compositions diminishes the concentration of monomer, leading to decreasing the polymerization rate of HEMA/PVP compositions and, naturally, improving their life time (Fig. 4, b).

As the results of previous researches [20, 23], with increasing concentration of polyvinylpyrrolidone and metal content in the initial composition the polymerization rate increases. Growth of the polymer-forming rate accordingly leads to reducing the life time of the initial composition (Fig. 5).

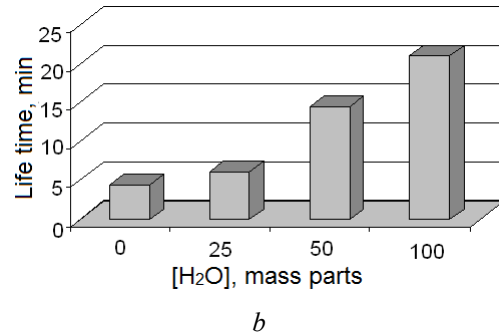
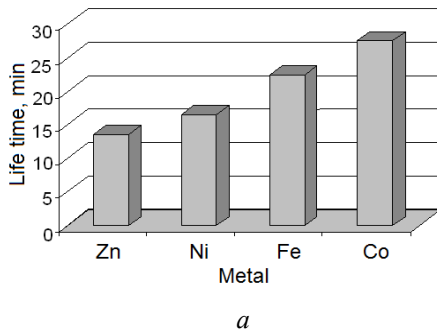


Fig. 4. Effect of the nature the metal (a) and solvent content (b) on the life time of HEMA/PVP compositions ($[\text{FeSO}_4] = 0,01 \text{ w. } \%$, $[\text{Me}] = 20 \text{ wt. } \%$, $MW_{\text{PVP}} = 12000$, $T = 293 \text{ K}$). Composition, m.p.: a – HEMA:PVP:H₂O = 70:30:50; b – HEMA:PVP = 70:30

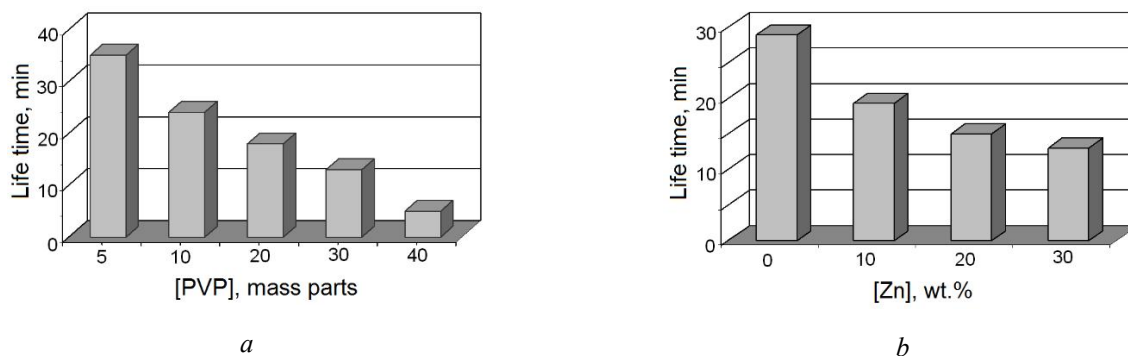


Fig. 5. Dependence the life time of the composition on the initial composition (a) and the content of the Zn powder (b) ($[FeSO_4] = 0,01$ wt. %, $[H_2O] = 50$ m.p., $MW_{PVP} = 12000$, $T = 293$ K): a – $[Zn] = 20$ wt. %; b – HEMA:PVP = 70:30 m.p.

3.3. The properties of the obtained composite metal film materials

Composite hydrogel film material samples obtained by centrifugal molding have attracted the attention by the isopachic not exceeding 1 % and surface quality [25]. For comparison, presented the surface of metal hydrogel sample obtained in the mold between the two polyethylene plates (Fig. 6, a) and by centrifugal molding (Fig. 6, b).

A similar composition was used for the processing of centrifugal molding (Fig. 6, b). The surface of the film, which is in contact with the forming side of mold, differs of homogeneity and high quality. The inner side (Fig. 7, a) is characterized

by significant inequalities of the surface. Diminishing the amount of solvent during centrifugal molding, measurable increased quality inner surface films (Fig. 7, b).

Research the results of the new composite metal hydrogels based on pHEMA-gr-PVP filled copolymer showed that the obtained materials have a variety of specific properties [26, 27], which may be regulated in a wide range by changing the initial composition and content of the solvent (Table 1).

Along with high water content and strength characteristics, it's primarily the presence of electrical conductivity, which has the ability to vary depend on the content of moisture.



Fig. 6. Effect of forming method on the surface quality of composite metal hydrogel film materials (HEMA:PVP:H₂O = 70:30:50 m.p., $[Zn] = 20$ wt. %): a – mold filling method; b – centrifugal molding



Fig. 7. Effect of solvent amount on quality of the metal hydrogel inner surface: a – $[H_2O] = 50$ m.p.; b – $[H_2O] = 25$ m.p.

Table 1

Effect of composite structure on physical and mechanical properties of the copolymers obtained in solution ($T = 293\text{ K}$, $MW_{\text{PVP}}=12000$, $[\text{FeSO}_4] = 0.01\text{ wt. \%}$, $\omega = 700\text{ rev./min}$, $[\text{Zn}] = 20\text{ wt. \%}$)

No.	Composition, mass parts		[H ₂ O], mass parts	σ_{br} , MPa	ϵ_{br} , %	W , %	k
	HEMA	PVP					
1	90	10	50	0.31	154	42	1.23
2	80	20	50	0.25	184	46	1.29
3	70	30	50	0.22	192	52	1.31
4	70	30	0	0.33	135	47	1.20
5	70	30	25	0.27	159	50	1.25
6	70	30	100	0.19	201	53	1.37

σ_{br} – tensile strength at break; ϵ_{br} – percentage elongation at break; W – water content, k – swelling coefficient

Table 2

Effect of the metal nature on the specific surface electrical resistance (ρ_s) and specific conductivity (γ_s) of copolymers obtained by centrifugal molding (HEMA:PVP:H₂O = 70:30:50 m.p., $[\text{Me}] = 40\text{ wt. \%}$, $\omega = 700\text{ rev./min}$)

No.	Filler (content in the composition)	ρ_s , Ohm	γ_s , Ohm ⁻¹
1	–	34.0×10^{10}	29.4×10^{-13}
2	Zn (10 wt. %)	15.2×10^9	65.8×10^{-12}
3*	Zn (20 wt. %)	$11.6 \times 10^8 / 41 \times 10^8$	$86.2 \times 10^{-11} / 32.3 \times 10^{-11}$
4	Zn (40 wt. %)	18.6×10^6	53.8×10^{-9}
5	Ni (10 wt. %) + Zn (10 wt. %)	26.5×10^7	37.7×10^{-10}
6	Fe (10 wt. %) + Zn (10 wt. %)	22.1×10^7	45.3×10^{-10}
7	Cu (10 wt. %) + Zn (10 wt. %)	18.4×10^6	54.4×10^{-9}

* The denominator – the electrical characteristics of the hydrogels obtained by mold filling method

These films are of interest in that the filler material, resulting in phase separation, distributed unevenly in thickness according to different surfaces characterized by different surface conductivity (Table 2).

Thus, the conductivity is notably higher on the outer surface and minimal on the inner surface. The specific surface conductivity of metal copolymers obtained by filling into molds (from the statistical distribution of metal particles) is way below than the surface conductivity of the materials obtained by centrifugal molding (Table 2, item. 3).

Conclusions

Consequently, the developed technology of centrifugal molding of metal filled composite hydrogel films characterized by a set of essential benefits and features compared to other formation

methods. Centrifugal technique let produce isopachic films with predetermined thickness and surface quality.

Using metal-containing compositions based on HEMA and PVP, choosing the nature, metal content and composite structure make possible to preset and change life time and duration of molding of compositions in the desired direction.

Ratio of HEMA:PVP and solvent content in the initial composition regulate the physical, mechanical, electrical properties and surface quality of hydrogel films in a wide range.

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О. М. Гриценко¹, І. Гайдос², Т. О. Гриценко¹, М. М. Яковенко¹

¹ Національний університет “Львівська політехніка”, кафедра хімічної технології переробки пластмас

² Технічний університет Кошице, кафедра технологій, матеріалів та автоматизованого виробництва

ТЕХНОЛОГІЧНІ ОСОБЛИВОСТІ ФОРМУВАННЯ ПЛІВКОВИХ ВИРОБІВ З МОДИФІКОВАНИХ ГІДРОГЕЛІВ ВІДЦЕНТРОВИМ МЕТОДОМ

Перероблення полімерів та композиційних матеріалів на їх основі методом відцентрового формування актуальне для виготовлення виробів у вигляді тіл обертання, які використовують у різних галузях народного господарства. В роботі проаналізовано відцентрове формування та подано результати дослідження нової сфери його використання як методу для виготовлення гідрогелевих плівок із матеріалів на основі кополімерів 2-гідроксіетилметакрилату з полівінілпіролідом, зокрема наповнених, які можна застосовувати у медицині, електротехніці та приладобудуванні.

Ключові слова: відцентрове формування; композиційні гідрогелі; кополімери; полівініл-піролідон; 2-гідроксіетилметакрилат.