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Chemistry

POLY(VINYL CHLORIDE)-POLYSTYRENE MATERIALS WITH POLYMER-SILICATE FILLERS

Volodymyr Levytskyi[1, 2], Diana Katruk[1], Andrij Masyuk[1, *],
Mykhailo Bratychak Jr.[1], Nataliia Chopyk[1]

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Abstract. The features of obtaining and modifying Ba- and Zn-containing polymer-silicate fillers have been researched. The influence of polystyrene modifiers (suspension and impact-resistant polystyrene) and finely dispersed Ba- and Zn-containing polymer-silicate fillers on the gelation process, view of flow curves, rheological properties: flow index, flow limit, thermal stability, surface hardness and elasticity of plasticized poly(vinyl chloride) have been discovered.

Keywords: poly(vinyl chloride), polymer-silicate filler, modification, gelation, polystyrene, polyvinyl alcohol, rheology.

1. Introduction

Application of materials based on plasticized poly(vinyl chloride) (PVC) and various inorganic fillers is caused by their valuable set of working properties as well as a wide range of compositions which appointed for the products of various purposes: in construction industry – linoleum, wallpaper, corrugated hoses, window profiles; in radio engineering – for isolation of cable products; in mechanical engineering – anticorrosive, sealing and insulating coatings, *etc*. Among the large number of inorganic fillers the silicate materials of various natures are considered perspective. Both natural silicate fillers (montmorillonite, kaolin, wollastonite, *etc*.) and synthetic ones are applied.[1]

Wide application of PVC products also takes place due to the variety of technological processes for the processing of plasticized PVC: calendering, extrusion, injection molding, *etc*.[2,3] All mentioned processing methods are accompanied by physico-chemical processes, which are primarily caused by the interaction of PVC macromolecules with a plasticizer and a filler.

It is possible to effectively affect the morphology of PVC materials and, thus, their properties due to the physical modification of described materials *via* additives of different nature, in particular polymeric[4,5] and inorganic ones.[6,7] Adding of mentioned additives allows to give PVC not only properties, which are inherent in separate components of a blend, but also new properties that, firstly caused by existence of interphase areas of various type. Physical modification of PVC based on adding of other polymers into the composition, is characterized by the influence of many physical and physical-chemical factors which are associated with the diversity of chemical and physical structure of polymers, interfacial characteristics of systems, intermolecular interactions, *etc.*

During the processing of polyvinyl chloride materials at elevated temperatures the great attention should be focused on increasing their resistance to thermal and thermal oxidative destruction.[8] Adding of inorganic nature additives allows not only to eliminate this disadvantage of PVC materials but also to increase the physical, mechanical and thermophysical properties. Currently an active use of heat stabilizers based on non-toxic mixtures of metal-containing compounds is being observed, in particular, based on Ba and Zn.

To give PVC plastics the necessary technological and operational characteristics along with other physico-chemical transformations, it is subjected to gelation. During gelling, the swelling of fine PVC with a constant

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[1] Lviv Polytechnic National University
12, S. Bandery Str., Lviv, 79013, Ukraine
[2] The John Paul II Catholic University of Lublin,
14, Al. Racławickie, Lublin, 20-950, Poland
[*] *masyukas@gmail.com*

decrease in the proportion of free plasticizer occurs. At the same time, the plasticizer is absorbed by PVC particles, increases their volume and as a result they are in direct contact with each other. As the temperature rises, the plasticizer penetration accelerates and the concentration of the plasticizer equalizes throughout the volume of the material. Fine PVC particles keep the boundaries of separation and with increasing temperature the monolithization of matrix occurs, which is accompanied by intermolecular interactions involving passage chains or their segments. During gelling the physical grid of PVC macromolecules is formed. The nodes of PVC grid include crystallites which exist at pretty sufficiently high temperatures within 483–503 K.

Physico-chemical processes (diffusion, absorption, swelling, *etc*.) that occur during the gelation of PVC plastics are significantly influenced by the temperature, component composition (primarily the presence of thermoplastic modifiers and inorganic fillers), heating duration, nature and plasticizer content. The completeness of the physical and chemical processes during gelling affects the choice of technological parameters and processing conditions of plasticized PVC materials, their rheological and operational characteristics.[9]

The aim of presented work was to determine the effect of Ba- and Zn-containing polymer-silicate filler and polystyrene modifier on physico-chemical processes occuring during the gelation of PVC plastics and their rheological and operational properties.

2. Experimental

The samples were prepared *via* the mixing of Lacovyl PB1156 brand PVC with a pre-dissolved polystyrene plastic (suspension polystyrene (SPS) or impact-resistant polystyrene (HIPS) in dioctyl phthalate (DOP) and metal-containing polymer-silicate filler.[10]

To obtain a polymer-silicate filler, an aqueous solution of sodium liquid glass (Na-LG) of 1 mol/L concentration and a modulus of n = 2.8 as well as a polyvinyl alcohol 16/1 (Nitrogen Production Association) and metals chlorides (Ba, Zn) have been applied. Scanning electron microscopy and elemental analysis of the obtained materials were conducted applying a REM-106I scanning electron microscope.

Gelation of PVC samples was carried out in a thermocouple 2B-131 for 1 h at 363 K. Rheological studies were conducted applying rotary viscometer Rheomat-30 in the shear rates range of 50–450 s^{-1} using a system of coaxial cylinders according to ISO 3219.

The pH determination of aqueous solution of PVC destruction products was made according to ISO 182-2.

Determination of viscoelastic characteristics was performed using a hardness tester TSHR-76.

Vicat softening point of the investigated materials was determined according to ISO 306: 2013, the load was 50 N.

The surface hardness of the conical fluidity point was determined applying Hepler Consistometer at 293 K by indentation of the steel zone in the polymer sample with a sharpening angle of $58^o08'$ under the load of 50 N for 60 s.

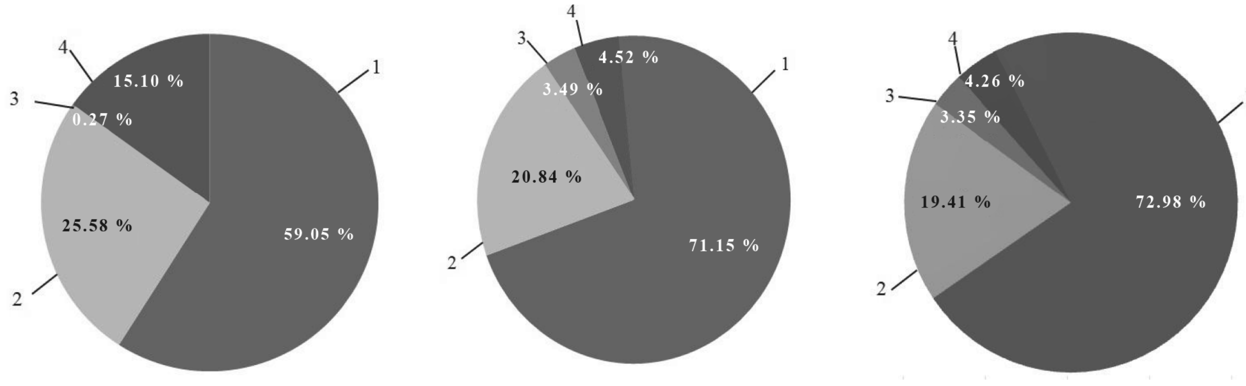
3. Results and Discussion

Finely dispersed Ba- and Zn-containing polymer-silicate fillers were rationally obtained on the basis of water-soluble silicates and polymers under the chlorides action of the corresponding metals. It is caused primarily through the possibility of regulating the morphology, technological characteristics of the filler as well as *via* environmental conditions of the process and the availability of domestic raw materials.[10]

At the same time, to increase the technological compatibility of the filler with a PVC polymer matrix it is advisable to carry out a preliminary modification of the fillers applying the combined deposition of water-soluble silicates and functionally active polymer. Such modification allows to obtain polymer-silicate composites containing the high molecular weight modifier which is evenly distributed in the silicate frame.[11] Polyvinyl alcohol (PVA) was applied as the polymer modifier.

To obtain Ba- and Zn-containing polymer-silicate fillers two ways were applied: according to the first one, Ba- and Zn-containing silicate fillers were obtained separately with further compatibilizing in a drum-type mill; as to the second one – co-precipitation of water-soluble silicate was carried out under the action of Ba and Zn chlorides blend.

It should be noted that adding of a polymer modifier into the reaction medium increases the precipitation efficiency. Based on energy-dispersion elemental analysis, it was found (Fig. 1) that in the case of materials obtained by physical mixing the individual particles are characterized by the predominance of one of the metals and insignificant presence of another. However, during co-precipitating the metals are equally distributed in silicate frame.

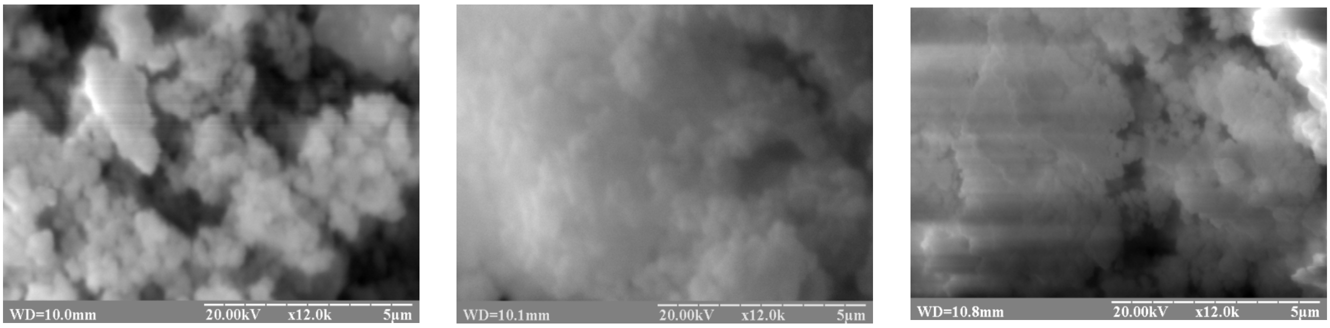


a) b) c)

Fig. 1. Elemental composition of the obtained metal-containing silicate materials, % at.: physical blend of Ba- and Zn-containing silicate fillers (a); co-precipitated Ba- and Zn-containing silicate filler (b) and co-precipitated Ba- and Zn-containing silicate filler modified with PVA (c). Elements: O (1); Si (2); Ba (3) and Zn (4)

The effect of polymer modifier on the elemental composition of obtained materials is insignificant. The oxygen content decrease relatively to silicon indicates the transition from the simplest partial silicate formations to the more complex ones (chain, belt, layered).[12] Above mentioned leads, respectively, to the decrease in the number of compensating cations (Ba and Zn), because on the border in frame structures where each of the four oxygen atoms of the tetrahedron SiO_4^- belongs simultaneously to two adjacent tetrahedral structures.

For determination of the morphological features of polymer-silicate composites the research applying scanning electron microscopy was also conducted. The obtained results are shown in Fig. 2.



a) b) c)

Fig. 2. SEM photographs of modified Ba- and Zn-containing silicate fillers: physical blend of Ba- and Zn-silicate fillers (a); co-precipitated Ba- and Zn-silicate filler (b) and modified with polyvinyl alcohol co-precipitated Ba- and Zn-silicate filler (c)

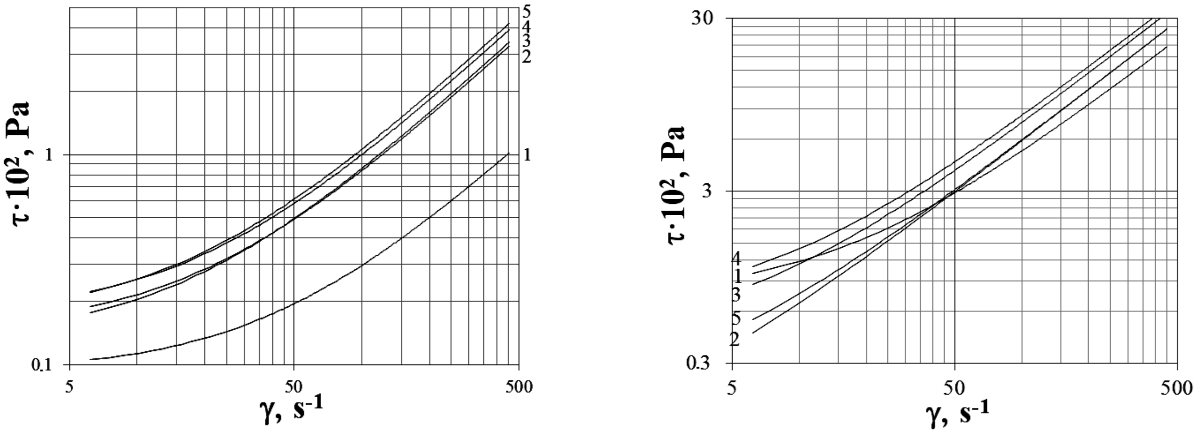
As one can see, the method of co-precipitation, compared with the method of simple physical mixing, leads to the formation of more homogeneous structures. At the same time, in the case of modified materials, the particles acquire a more regular oval shape and possess smaller dimensions. It should be noted that polyvinyl alcohol application leads to the formation of agglomerates contained inclusions of 50–200 nm.

Based on sorption studies, it was found that modified silicate fillers have a much lower value of moisture absorption (by 15–25 %) than unmodified ones. In this case, the obtained silicate fillers are characterized by significant values of available active surface area in relative to the alkali-acid indicator of methylene blue (60–75 m^2/g). Modification with polyvinyl alcohol causes some reduction of above-mentioned values. This is, obviously, explained by the blocking with PVA macromolecules of the surface groups of silicate formations as well as by coating of the surface of the particles with hydrophobic carbon chains.[13]

We have created Ba- and Zn-containing polymer-silicate materials and used them as fillers in PVC. The resulting PVC-based composites have higher thermal stability than unfilled PVC. Also rheological, physico-mechanical and thermophysical properties of such composites are better than those of the starting PVC.

Viscosimetric research was conducted to determine the effect of Ba- and Zn-containing polymer-silicate materials as well as the gelling process on the rheological properties of PVC plastics.

Since PVC plastics are processed in a wide range of stresses and shear rates, the correlation of which characterizes the impact on the technological characteristics of the material during processing.[14] As a result, the dependencies were built in the form of flow curves of plasticized PVC materials depending on nature of polystyrene modifier and gelling conditions which are given in Fig. 3.



a) b)

Fig. 3. Flow curves of the filled modified PVC plastics: before (a) and after (b) the gelation stage. The compositions content: PVC:DOP = 20:80 (1); PVC:SPS:DOP = 16:4:80 (2); PVC:SPS:DOP:PSF = 15:4:80:1 (3); PVC:HIPS:DOF:PSF = 15:4:80:1 (4) and PVC:HIPS:DOP = 16:4:80 (5)

Adding of polystyrene modifiers, regardless of its nature, leads to an increase in the values of shear stress. After the gelation stage the values of shear stresses increase regularly. As we can see, the nature of the flow curves of modified materials, which are not gelled, is similar to each other in both the non-Newtonian fluid and Newtonian flow area. It should be noted that for unmodified compositions which have been subjected to gelation, the nature of the flow curves is similar to the gelled compositions. For the modified compositions, significant changes are observed, especially in the field of shear rates up to 50 s^{-1}. This is obviously due to the fact that adding of polystyrene modifiers reduces the internal friction and shifts the beginning of viscosity anomaly. The less appreciable detection of the viscosity anomaly for gelled modified plastics is most likely caused by the redistribution of intermolecular interactions between macromolecules of PVC and modifier during gelling as well as by the the formation of a fluctuation grid, the nodes of which have PVC crystallites are replaced by the modifier macrochain segments.

The nature of the flow curves of materials based on PVC plastics, which are multicomponent systems, is also influenced by the adding of fine additives, in particular Ba- and Zn-containing PSF, which will play the role of thermal stabilizers in the mentioned materials. The nature of the flow curves of the modified PVC materials filled with PSF, which were subjected to gelation, does not depend on the polystyrene modifier nature. After the gelling stage, there is an increase in the viscosity of the filled modified PVC plastics. It should be noted that the materials modified by the HIPS are characterized by higher values of shear stress.

It is known[15] that the Caisson model allows to characterize the rheological nature of the behavior of polymer-containing system, which is determined by the following mechanisms: the collapse of the spatial structure, the destruction of smaller structural elements and the orientation of asymmetric aggregates. The parameters of the Caisson model reflect the structural properties of the system; the shear stress is associated with the primary decay of the spatial structure grid, and the Caisson viscosity characterizes the state of the system during the orientation of asymmetric units.

According to the above, the flow characteristics of polymer materials are characterized by a flow index *n*, which is determined from the graphical dependence $\lg\tau - \lg\gamma$. The values of yield strength ($\tau_{gr.}$) and Caisson viscosity (k) are determined from the graphical dependence $\tau_2 = f(\gamma_2)$. The obtained values of *n*, τ_{gr} and *k* for modified PVC plasticates are given in Table 1.

Table 1. Rheological parameters of modified PVC plastics

Composition content, parts by weight					*n*	$\tau_{gr.}$, Pa	*k*, Pa·s
PVC	DOP	SPS	HIPS	PSF			
20	80	–	–	–	0.61* 0.84**	9.3 73.2	0.14 3.43
16	80	–	4	–	0.84 1.00	13.2 39.5	0.61 5.83
15	80	–	4	1	0.87 0,91	17.1 62.8	0.76 7.00
16	80	4	–	–	0.86 0.97	14.6 42.6	0.57 6.41
15	80	4	–	1	0.88 1.00	16.6 18.6	0.73 5.48

Notes: * – before the gelation stage; ** – after the gelation stage.

After the gelation stage, the modified PVC compositions are characterized by an increase in the value of the flow index, regardless of polystyrene modifier nature. The nature of PVC plastics flow modified with both HIPS and SPS, as well as those containing PSF, is marked by the flow index ≈1, which indicates the Newtonian nature of the flow. The flow index increase is apparently due to the formation of a special intermolecular structure between the macromolecules of PVC and the polystyrene modifier. In this case, the change in the nature of the flow of PVC compositions, apparently, is influenced by a number of physico-chemical factors, which are caused by adding the modifiers of different nature and greater mobility of macromolecule segments at elevated temperatures during gelling. During the gelation stage, the macromolecules of polystyrene modifier penetrate into the swollen PVC agglomerates due to the influence of temperature leading to morphological changes of PVC.

The flow of polymer materials is characterized by the yield strength, which is a consequence of the spatial fluctuation grid associated with Van der Waals, electrostatic and polar interactions between macromolecules and other components. The lower yield strength, the lower the consumption that is required to bring the material to a fluid state and support the material flow at the level required for processing.

It was found that before the gelation stage, the modified PVC compositions are characterized by higher values of yield strength than the unmodified composition. It was found that after the gelation stage, all modified PVC compositions are marked by an increase in the yield strength. After the gelation step, the modified PVC compositions are characterized by lower yield strength values compared to the unmodified PVC composition. Such changes in the yield strength of modified PVC compositions may be related to the effect of polystyrene modifier macromolecules on the swelling of PVC macromolecules during the gelation stage, as well as filling the volume formed by the decay of PVC agglomerates with macromolecule segments.

The determined dependences of the change in the yield strength of PVC compositions modified with both thermoplastic polystyrene additives and PSF allow to predict changes in the technological parameters of processing of plasticized materials based on PVC.

Adding of inorganic additives as thermal stabilizers eliminates a significant disadvantage of PVC materials – their low heat resistance and, consequently, the complication with processing, as the temperature of transition to a viscous state is close to the temperature of PVC destruction. Today, there is a replacement of harmful lead-containing thermal stabilizers with non-toxic mixtures of metal-containing compounds based on Ca/Zn, Ba/Zn and Ca/Ba/Zn.[16]

Based on potentiometric studies of PVC degradation products, the influence of the nature of polystyrene modifier and PSF on the thermal stability of PVC plastics was determined. The results of aqueous solutions potentiometric titration of degradation products of modified polyvinyl chloride materials are shown in Fig. 4.

It is determined that in the presence of a polystyrene modifier, both SPS and HIPS, the thermal destruction of PVC material is being suspended. It should be noted that in the case of SPS application the resistance to a thermal destruction is higher. Metal-containing PSF pre-modified with polyvinyl alcohol, significantly increases the thermal stability of the modified PVC material that is especially evident at temperatures above 463 K.

The slowing down of destructive processes in modified materials may be associated with a denser fluctuation grid formation at the direct participation of macromolecules of the polystyrene modifier and, as a consequence, the prevention of hydrogen chloride release. PVC plastics, which contain Ba- and Zn-containing PSF, possess the highest thermal stability. Obviously, this is due to exothermic processes of interaction of PVC degradation products (primarily hydrogen chloride) with Ba- and Zn-containing PSF which may be associated with blocking of thermal destruction centers in PVC by an active surface groups of silicate filler formations. Also, it is caused by its interaction with products of destruction as well as lower density of the fluctuation grid due to the presence of PSF and via the transient adsorption layer formation near its particles.

Conducted thermomechanical studies allow us to conclude about the value of deformation in the low-temperature range for PVC-PS plastics filled with PSF, compared with unfilled (Fig. 5).

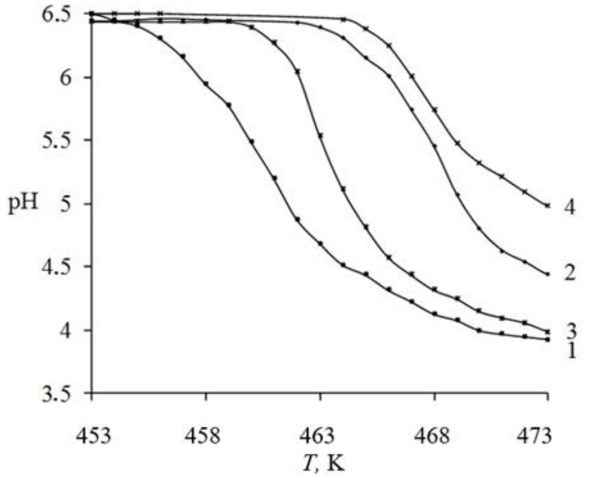


Fig. 4. Influence of polystyrene modifier and Ba- and Zn-containing PSF on the value of HCl release during PVC thermal destruction. The content of polystyrene modifier is 2.8 parts by weight: none (1); SPS (2); HIPS (3); Ba- and Zn-containing PSF (4)

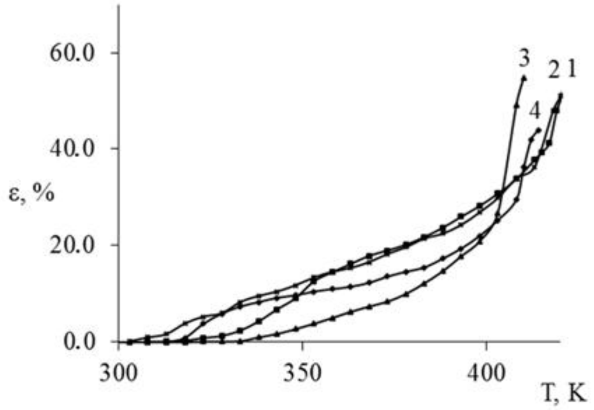


Fig. 5. Influence of PSF on thermomechanical properties of polystyrene-modified PVC plastic (PVC: DOP:SPS = 100:30:2.8). Filler, weight parts per 100 wt. of PVC: without PSF (1); Ba- and Zn-containing PSF, 1 wt. part (2); Ba- and Zn-containing PSF, 2 wt. parts (3) and Ba- and Zn-containing PSF, 10 wt. parts (4). PSF modifier: 3, 4 - PVA (0.5 wt %)

In this case, modified by polyvinyl alcohol PSF significantly affects the thermomechanical properties of PVC composites. At a temperature of 403 K PSF promotes more intense mobility of the segments, mutual movements of macromolecules under the action of load are possible. Above mentioned indicates the transition of the polymer composition from a highly elastic state to a viscous one. In this state, the mobility of kinetic elements is realized: individual atoms and groups, segments and macromolecules. It should be noted that materials containing PSF modified with polyvinyl alcohol are characterized by increased deformation at slightly lower temperatures. This indicates that under the action of the modified PSF the changes in the fluctuation grid occurred. It leads to a faster softening of the polymer material. The low content (1 % wt.) of unmodified PSF does not affect the thermomechanical characteristics of PVC-PS materials.

It was found that Ba- and Zn-containing PSF also affects the thermophysical and physico-mechanical properties of modified PVC plastics: the number of elasticities (*S*) and surface hardness (*F*) (Table 2).

Table 2. Influence of Ba- and Zn-containing PSF on physico-mechanical and thermophysical properties of PVC plastics (PVC:DOP:SPS = 100:30:2.8 (parts by weight)

PSF content, parts by weight	*S*, %	*F*, MPa	*Tv*, K
0	59.0	80.4	312
2	17.9	73.8	325
2*	80.4	108.1	322
10*	61.7	93.3	333

Note: PSF modified by poly(vinyl) alcohol

Adding of of PSF modified with polyvinyl alcohol (2 %) causes an increase in the value of surface hardness by ≈ 30 MPa and the number of elasticities by ≈ 20 %. At the same time, the unmodified PSF reduces the value of surface hardness and elasticity, which is obviously caused by the greater heterophase of the system due to the lower technological compatibility of the filler and the polymer matrix. It should be noted that at 10 % of the modified Ba- and Zn-containing PSF some decrease in the value of the elasticity and the maximum value of Vicat softening point is being observed.

4. Conclusions

The new Ba- and Zn-containing polymer-silicate fillers have been developed and their properties have been determined. The influence of surface-active polyvinyl alcohol on the process of metal-containing silicate fillers derivation as well as its morphology and properties are revealed.

The influence of polystyrene modifier nature (impact-resistant and suspension polystyrene) on the physico-chemical regularities of PVC plastics gelation is defined. It was found that SPS increases the viscosity of plasticized PVC compositions during gelling to a greater extent than HIPS, which is a consequence of the redistribution of intermolecular interactions with the formation of a denser fluctuation grid at the participation of heterogeneous macromolecules. It was found that the polystyrene modifier and fine polymer-silicate filler affect the nature of the flow curves of PVC compositions as well as the rheological parameters of the modified PVC plastics.

The influence of polymer-silicate filler and polystyrene modifier on physico-mechanical and thermophysical properties of PVC plastics has been determined. Adding of a silicate filler modified with polyvinyl alcohol to the composition allows to increase the value of surface hardness by ≈ 30 MPa, the number of elasticities by ≈ 20 % and Vicat softening point as well as to increase the resistance of PVC composites to thermal destruction.

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ПОЛІВІНІЛХЛОРИД-ПОЛІСТИРОЛЬНІ МАТЕРІАЛИ З ПОЛІМЕР-СИЛІКАТНИМИ НАПОВНЮВАЧАМИ

***Анотація**. Виявлено особливості одержання і модифікування Ba-, Zn-вмісних полімер-силікатних наповнювачів. Встановлено вплив полістирольних модифікаторів (суспензійного і удароміцного полістиролу) та дрібнодисперсних Ba-, Zn-вмісних полімер-силікатних наповнювачів на процес желювання, характер кривих течії, реологічні характеристики: індекс течії і границю текучості, а також на термостабільність, поверхневу твердість і число пружності пластифікованого полівінілхлориду.*

***Ключові слова:** полівінілхлорид, полімер-силікатний наповнювач, модифікування, желювання, полістирол, полівініловий спирт, реологія.*