Rheological analysis of PVC plastisols containing a surfactant, an organic pigment, a thickener and water

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Introduction

Plastisols of poly(vinyl chloride) (PVC) are dispersions of powder polymer in a plasticiser and are modern surface materials, which do not require transforming the polymer into an enamel solution. Plastisol based protective enamels are characterised by high resistance to mechanical damage and chemically aggressive conditions [1].

One significant drawback of plastisols, however, is their low stability, which results in changing their technological properties during preparation, application and storage. In general it can be said, that substance viscosity increases and sedimentation connected to phase separation in the plastisol can be observed, which results in the formation of polymer prescipitate. Increasing viscosity can be attributed to the plasticiser being absorbed by PVC grain and the accumulation of molecules thereof [2-4].

Plastisol molecules may be characterised by their micellar structure. This is a result of thermodynamic properties of such systems and excess free surface energy on the expanded interphase surface. Therefore such systems may undergo spontaneous molecular aggregation. A colloidal plastisol molecule acts as a micelle built of a polymer-rich nucleus and an external solvatative layer containing both the polymer and plasticiser. It is furthermore obvious, that processes taking place within the solvatative layer and between them influence flocculation and coalescence of PVC plastisol molecules [5,6].

Another method of evaluating plastisol stability is the analysis of particle size fluctuations and their arrangement regularity, within the dispersive phase, as a function of time. This method is also based on measuring viscosity using various measuring equipment. In most cases polymeric dispersive systems display non-Newtonian flow behaviour. The main reason for viscosity changes of the plastisol during flow is the fact that the structure of such fluid is subject to reversible change. The network of inner intermolecular bonding is destroyed, the more, the larger the shear stress, subsequently undergoing spontaneous regeneration to their initial state. Particle associates, created through the network decomposition, increase in energy through this process, and later lose this excess energy while reforming the previous internal structure. As a result the number of polymer segments comprised of newly formed particle associates increases, which in turn leads to decreasing the structural viscosity of the system. However evaluating the exact role each individual factor plays on the flow of dispersive systems based on rheological studies is not yet possible [7].

The Faculty of Protective Coating Technology of UTLS has been conducting research for many years into producing aqueous dispersions of PVC plastisols intended for use in protective coating. It was determined that introducing a liquid organic additive to such systems results in decreasing PVC plastisol particle size and increasing stability of its aqueous dispersion [8].

Poly(vinyl chloride) plastisol protective coatings are obtained using immersion coating or using roll-to-roll processes previous to gelation. Introducing an organic diluent into the plastisol allows for decreasing its viscosity and the obtained product can be applied using hydrodynamic methods. These methods can be used to increase corrosion resistance in roofing materials or produce waterproof coated fabrics [9,10].

The aim of the research presented in this work is the evaluation of the influence of additive surfactants, organic pigments, thickeners and water on flow characteristics. It was assumed, that the plastisol dispersive system is a non-ideal composition, due to the fact, that polymer grains undergo various degrees of solvatation or swelling due to the presence of the plasticiser, and subsequently cooperate. The surfactant, organic pigment, thickener or water introduced may induce or favour the formation either of swollen PVC grain associates or a network of internal bonds. The possibility of introducing the mentioned additives into the plastisol allows for speculation of dispersing it in water and obtaining a stable dispersion. Using such a method the plastisol could be applied by pneumatic spraying. Scientific literature thus far lacks a full description of the research endeavour undertaken, which persuaded us to provide an explanation of it.

Experimental proceedings

Materials

The plastisols were prepared using poly(vinyl chloride) type PVC E-68 Pmbs (K=69.5) made by Zakłady Chemiczne "Synthos Dwory" Sp. z o.o. in Oświęcim. The plasticiser was di-n-buthyl phtanal (DBP) of molecular mass 278.34 g/mol, density 1.043 g/cm³, molar refractivity

Table 1

Selected physicochemical properties of surfactants used during research

Chemical name and manufacturer designation	Molecular mass M, g/mol	Hydrophilic-lipophilic balance HLB	Critical micellar concentration $C_{CMC}*10^3$ mol/dm³	Surface activity G pow.*10^{-3}, mN/m*dm³/mol	Critical viscosity number GLL, cm³/g	Haller constant k	Hildebrandt solubility parameter ä, [cal/cm³]^0,5
Rokanol L-10	610	14,4	0,8	47,8	0,0013	0,17	8,4
Rokanol O-18	1013	15,6	0,30	74,3	0,0017	0,17	6,1
Rokanol L-25	1300	17,0	0,20	86,0	0,0054	0,11	8,3
Rokacet S-24	1326	16,0	0,40	68,1	0,0014	0,15	8,3
Rokamin SR-22	1227	15,8	0,08	68,3	0,0016	0,18	6,5
Rokamid MT-17	1059	14,1	0,09	61,4	0,0013	0,16	6,8

Selected properties of organic pigments used

Chemical name and manufacturer designation	Pigment density g/cm³	Apparent volume dm³/kg	Specific surface area m²/g	Oil absorption number g/100g
Iron (III) and 1-nitrosonaphtalen-2 (BO Green)	2,02	0,49	35,0	46,0
2-Dichloroaniline-3-hydroxy-2-naphtalene-o-methyl anilinoether (RLL Red)	1,44	0,69	18,0	42,0

R=77.12 cm³/mol, dipol moment 0.912 and *Hildebrand* solubility parameter 20.12x10³[J/m³]$^{1/2}$, which in turn is produced by Zakłady Chemiczne "Boryszew-Erg" in Sochaczew. Surfactants were manufactured by Zakłady Chemiczne PCC "Rokita" S.A. in Brzeg Dolny. Selected physicochemical properties of those substances are presented in Table 1.

Rokanol L-10 is oxyethylenated lauryl alcohol, Rokanol O-18 is oxyethylenated unsaturated fatty alcohol, Rokanol L-25 is oxyethylenated lauryl alcohol, Rokacet S-24 is oxyethylenated stearic acid, Rokamin SR-22 is oxyethylenated fatty amine, Rokamid MT-17 is oxyethylenated ethanoloamide. Chemical name and molecular mass SPC were supplied by the manufacturer in the material safety data sheet. PVC plastisols were dyed using dry organic pigments, which were BO Green and RLL Red made by Wolskie Zakłady Przemysłu Barwników "Organika" in Wola Krzysztoporska. Table 2 shows a selection of their characteristics taken from the manufacturer's material safety data sheet.

The thickener used was a water soluble polyurethane oligomer "Schwego PUR 8050", manufactured by Schwegomann, of active ingredient content 17.5% weight, pH of 2% solution 6-8, density 1.07 g/cm³.

Plastisol sample preparation

The plastisols used contained 100 parts by weight PVC and 120 parts by weigh DBP. The samples were prepared by thorough mechanical mixing of PVC powder and the plasticiser in a mortar over the course of 8 h. Plastisols were subsequently rested and deaerated in a vacuum dryer (type HZV) under 6.5 hPa pressure and in 22°C temperature for 48h. Samples were introduced for study 24h after deaeration. Plastisols were homogenous, did not delaminate and contained no residue. A detailed description of obtaining plastisols can be found in sources [1, 4]. Plastisol samples of 25 g were introduced with a fixed amount- 2.0 g of surfactant, which constituted 7.4% weight and thoroughly stirred. Afterwards samples thus obtained were introduced, during mortar homogenisation, with water of: 10.0; 18.0; 27.0; 30.0; 36.0; 48.0 and 65.0% weight. For each surfactant seven water containing plastisol samples were prepared. These samples were then in turn subject to rheological analysis. To evaluate the influence of or-

ganic pigment and water contents on plastisol rheological properties containing a fixed amount of surfactant, a set of samples was prepared containing 25 g of PVC plastisol and 0.5 g of surfactant (2% weight). In this case Rokanol L-10 and Rokanol O-18 were used. The amount of organic pigment in each plastisol sample was: 0.5; 1.0; 3.0; 5.0; 8.5 and 12.0% weight. Respectively the amount of water added to each mentioned plastisol sample containing a fixed amount of surfactant and fixed amount of pigment was: 0.02; 0.054; 0.12 and 0.2% weight.

To evaluate the influence of thickener on rheological properties of a plastisol composition, samples were prepared using a method similar to the evaluation of the influence of variable organic pigment and water concentration. In this case, however, the thickener content was fixed and was 1.5% weight in each analysed plastisol sample.

Evaluation methods

The viscosity of test plastisol composite samples was measured using a *Höppler* rheoviscosimeter type 202 made by MLM Prüfgeräte-Werk Medingen/Sitz Freital in 20±0.1°C using shear stress ranging from 10 to 150 mPa. The dependence of viscosity on shear stress is described by *Ostwald – de Wael* exponential equation in the form of (1) [4,7] :

$$\eta = k \cdot P^{m-1} \qquad (1)$$

where k Pa^{2-m}s is a consistency parameter, m is plasticity parameter and P is shear stress. Results were interpreted using the modified equation (2):

$$\eta = k \cdot P^{-n} \qquad (2)$$

which was used to calculate k constant using the experimental data represented as a straight line in a $\lg\eta = f(\lg P)$ coordinate system. By extrapolating $\lg P \to 0$ for the value in which the line crosses the $\lg\eta$ axis, the logarithm of viscosity is found, which is then used to calculate the value of k. In this case k is called maximum viscosity and denoted by η_0, n=1-m is the plasticity coefficient. Activation energy of viscous flow for the plastisol composition was calculated using *Arrhenius and Guz-*

Constants describing dependencies of maximum viscosity, pseudoplasticity coefficient, and viscous flow activation energy for a plastisol containing a constant surfactant concentration and increasing water content

| Surfactant type	Constant type	First range of plastisol water content from 0 to 30%weight.			Second range of plastisol water content from 30 to 65%weight.		
		$\lg\eta_0$	n	E_η	$\lg\eta_0$	n	E_η
Rokanol L-10	a	0,0057	0,0016	0,0597	0,0523	0,0067	0,579
	b	3,75	0,411	37,756	5,235	0,5782	54,711
Rokanol O-18	a	0,0074	0,0008	0,1316	0,0483	0,0037	0,482
	b	4,7137	0,3901	40,321	5,9342	0,4817	50,924
Rokanol L-25	a	0,0141	0,0006	0,1064	0,0473	0,0039	0,4802
	b	4,4369	0,3874	38,998	5,4759	0,502	50,477
Rokacet S-24	a	0,0117	0,0008	0,0836	0,0471	0,004	0,5666
	b	4,4882	0,4282	45,824	5,6552	0,5346	61,608
RokaminSR-22	a	0,0085	0,001	0,1316	0,0614	0,0067	0,6838
	b	4,3171	0,44	49,052	6,001	0,6281	66,496
Rokamid MT-17	a	0,0143	0,0014	0,0869	0,0343	0,0019	0,4093
	b	3,9232	0,2924	35,872	4,5344	0,2999	45,934
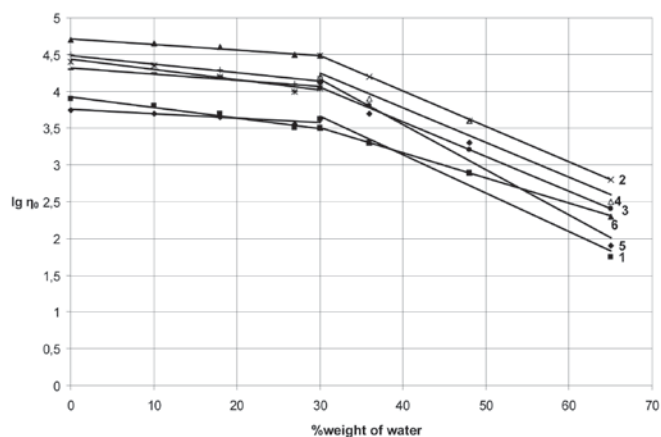
Fig. 1. Dependence of maximum viscosity of PVC plastisols with set surfactant concentration on water content

Description: 1 – Rokanol L-10.2 – Rokanol O-18,
3 – Rokanol L-25,4 – Rokacet S-24,5 – Rokamin SR-22,6 – Rokamid MT-17
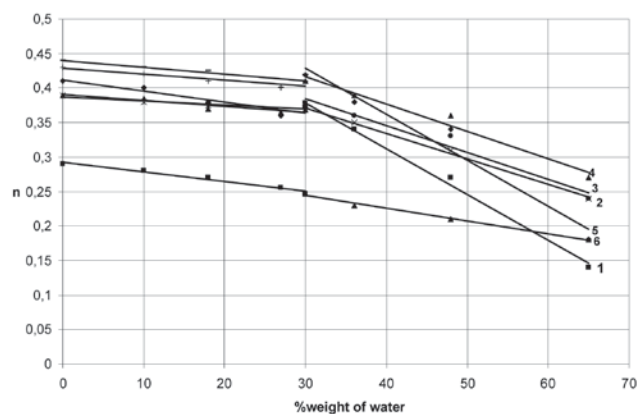

Fig. 2. Pseudoplasticity factor dependence on water content in a plastisol of constant surfactant concentration

Description: 1 – Rokanol L-10,
2 – Rokanol O-18, 3 – Rokanol L-25, 4 – Rokacet S-24, 5 – Rokamin SR-22, 6 – Rokamid MT-17
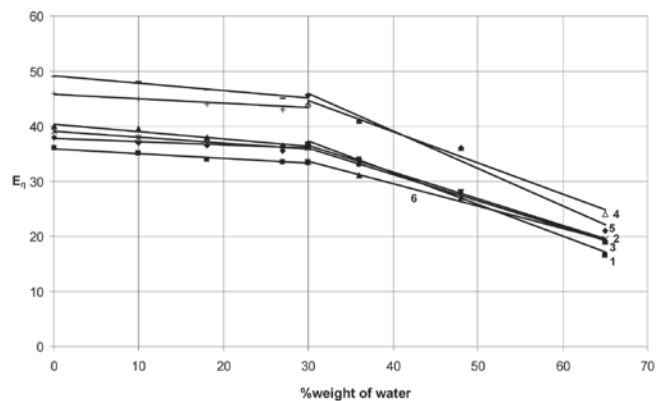

Fig. 3. Activation energy of viscous flow dependence on water content in a plastisol of constant surfactant concentration

Description: 1 – Rokanol L-10,
2 – Rokanol O-18, 3 – Rokanol L-25, 4 – Rokacet S-24, 5 – Roakmin SR-22, 6 – Rokamid MT-17

man equation [2,7]. To achieve three measurements of viscosity were conducted at 20, 25, 30, 35 and 40°C using constant shear stress of 20 mPa. Activation energy for viscous flow was calculated taking into account the average viscosity value at given temperature. All calculations were conducted using Microsoft Excel spreadsheet, as part of Windows XP software.

Test results and their description

One of the more unique properties of plastisols is their viscosity. The plastisol itself is a composition of compounds of very com-

plex structures and large range of particle size. Interactions between plastisol constituents may change according to changing mechanical stress exerted on it. Rheological analysis allowed for evaluating a flow curve as viscosity dependence on shear stress. Based on the curve shape it was ascertained that the plastisols tested behave in a manner characteristic for typical pseudoplastic fluids, characterised by decreasing viscosity with increasing shear stress. It can be assumed that such behaviour can be explained by plastisol particle deformation, particle associate decomposition, and the disintegration of internal network of intermolecular bonds. Using the obtained flow curves and based on equation (2) the following parameters were determined: maximum viscosity, pseudoplasticity coefficient, and using Arrhenius and Guzman equation, the activation energy of fluid flow was calculated. Fig. 1, 2, 3 show the dependence of changes in maximum viscosity, pseudoplasticity coefficient and activation energy of fluid flow for a plastisol containing a constant amount of surfactant and increasing water content.

Dependencies shown in Fig. 1, 2 and 3 can be described by straight line equations taking into account two separate ranges for plastisol water content. The first range corresponds to water content between 0 and 30% weight and second range - 30 to 65% weight:

$$\lg \eta_0 = \left(a \cdot c_{pig} + b\right) - \left(c \cdot c_{pig} + d\right) \cdot c_{H_2O} \qquad (3)$$

$$n = \left(a \cdot c_{pig} + b\right) - \left(c \cdot c_{pig} + d\right) \cdot c_{H_2O} \qquad (4)$$

$$E_\eta = \left(a \cdot c_{pig} + b\right) - \left(c \cdot c_{pig} + d\right) \cdot c_{H_2O} \qquad (5)$$

where c_{H2O} is water content in the plastisol composition in % weight.

Constants for equations (3) – (5) are found in Table 3.

Based on data shown in Table 3 it can be stated, that in a plastisol of constant surfactant concentration and increasing water content a phase inversion phenomenon takes place. The schematic below shows the hypothetical mechanism for such a phenomenon.
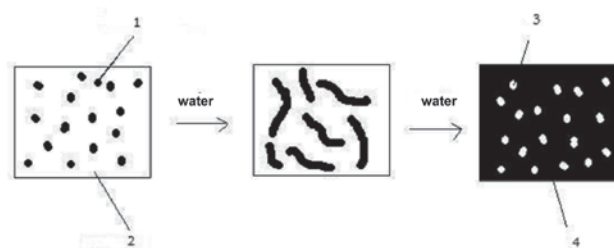

Fig. 4. Schematic representation of phase inversion phenomenon in a plastisol containing a surfactant and water

Description: 1 – water- dispersed phase containing surfactant, 2 – PVC plastisol dispersive phase, 3 – dispersed phase- plastisol molecules stabilised by surfactant, 4 – water- dispersive phase

Phase inversion phenomenon occurrence depends on changes of the dispersive system type. Observing this phenomenon in plastisols was possible due to viscosity measurements. In the plastisol the initial dispersed phase becomes the dispersive medium and the initial continuous phase begins forming micelles. This process increases in intensity while the water content increases in the plastisol, while smaller micelles begin to connect. The phenomenon of formation and connecting of water micelles happens in the first water content range. At 30% weight content of water in plastisol the phase inversion takes place. From this moment on water micelles begin to connect to form one homogenous dispersive phase, while forming a dispersed phase of plastisol stabilised by the surfactant. During the inversion several phenomena can be distinguished- the formation of water micelles containing the surfactant in the plastisol environment, their coalescence and the dissolution of water floccules with formation of dispersed plastisole particles characterised by adsorptive layers of surfactant.

Subsequent studies were concerned with determining changes of flow parameters for a PVC plastisol containing a fixed amount of surfactant and a variable concentration of organic pigment and water. Two non-ionic were chosen for the study, differing significantly in ethoxylation number, and organic pigments differing in specific surface area and oil absorption number. Based on tests conducted it became apparent, that plastisol compositions behave in a manner characteristic for pseudoplastic and their flow curves allow for determining the logarithm of maximum viscosity, pseudoplasticity coefficient and allow for calculating the activation energy of viscous flow. The results obtained were elaborated on mathematically. A dependence was obtained in the form of equations shown below:

$$\lg \eta_0 = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (6)$$

$$n = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (7)$$

$$E_\eta = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (8)$$

where c_{pig} is pigment concentration and c_{H2O} is water concentration in %weight.

In equations (6) ÷ (8) the amount of organic pigment varied within range from 0.5 to 12.0% weight, while the amount of water added to each plastisol sample containing pigment ranged from 0.02 to 0.2% weight. The amount of surfactant was equal 2.0%weight and was constant for all samples. Table 4 contains values for constants present in the described equations.

Based on data shown in Table 4 it can be stated, that introducing BO green into the plastisol results in a larger increase in viscosity than the introduction of RLL Red. Meanwhile the addition of water to plastisols containing the same amount of pigment resulted in a more drastic viscosity reduction, when the pigment in question was BO Green, and not RLL Red. It turns out, that using the same amounts of surfactants we obtained lower maximum viscosity, pseudoplasticity coefficient and viscous flow activation energy, when the plastisol in question contains Rokanol L-10 than Rokanol O-18. It stems from the fact, that Rokanol L-10 has a lower ethoxylation number and weaker surface capabilities.

Based on studies performed the following mechanism can be proposed for the flow of an organically dyed plastisol containing a surfactant and water. Within the bulk of the plastisol water micelles form, containing the surfactant inside. The organic pigment is spread evenly throughout the plastisol. The apparently larger viscosity of plastisols containing BO Green over RLL Red is caused by the fact, that BO Green has a bigger specific surface area and absorbs the plasticiser from the plastisol bulk much more effectively. This dependence is shown in the first part of each of the presented equations. Water in

turn influences rheological properties of plastisols by reducing viscosity with its addition. The viscosity drop in this case is more significant for Rokanol O-18 than Rokanol L-10. The size of water and surfactant micelles depends on the amount of water, and especially on the degree of hydrophilisation of the surfactant. Water micelles containing Rokanol O-18 are larger and exhibit stronger surface properties and therefore a more significant ability to lower the viscosity of a plastisol composition. This dependence is in turn described by the second parts of presented equations. In these systems, the emergence of micelle associate particles is not impossible.

Subsequent studies pertained to determining the influence of a constant concentration of a thickener on rheological parameters of PVC plastisols containing a set amount of surfactant and variable concentrations of pigment and water. These were conducted on compositions identical to those presented above. They contained a fixed amount of either Rokanol O-18 or Rokanol L-10 equal to 2.0%weight and will now additionally contain a constant amount of 1.5% weight of a water-soluble polyurethane oligomer. The rheological analysis conducted thus far showed, that the plastisols remain pseudoplastic fluids; their flow curves can be used to determine maximum viscosity, pseudoplasticity coefficient and to calculate activation energy of viscous flow. The results obtained were evaluated mathematically resulting in the following equations:

$$\lg \eta_0 = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (9)$$

$$n = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (10)$$

$$E_\eta = \left(a \cdot c_{pig} + b\right) \cdot c_{H_2O} + \left(c \cdot c_{pig} + d\right) \qquad (11)$$

Table 5 shows constant values pertaining to each rheological parameter describing the flow of analysed plastisol compositions.

Analysing equations (9) – (11) and the data shown in Table 5 it can be clearly seen, that increasing the concentrations of pigments and water in a plastisol containing a constant amount of surfactant and thickener results in increasing maximum viscosity, pseudoplasticity factor, and activation energy of viscous flow. In this case the addition of a thickener resulted in a situation different than when no thickener was present. Data obtained shows significant increases in rheological parameters, when the composition contains BO Green and Rokanol O-18. In all plastisol systems containing the thickener analysed, the introduction of water resulted in increased viscosity of the composition. It would seem that the thickener presence within the system results in the water introduced to cause significant hydration of micelles made up of the surfactant and thickener. In such conditions the thickener creates a network of bonds from the micelles, which is resistant enough to decomposition or deformation under shear stress that it results in

Values of constants for equations (6)– (8) describing changes in maximum viscosity, pseudoplasticity coefficient and activation energy of viscous flow of plastisols containing a fixed amount of surfactant and variable concentration of pigments and water

Rheological parameter	Pigment type	Surfactant type	Constant type			
			a*10	b	c*10²	d*10
Maximum viscosity lg η₀ mPas	BO Green	Rokanol L-10	1,6	3,4	0,50	0,16
		Rokanol Ł-18	1,9	3,6	0,59	0,18
	RLL Red	Rokanol L-10	1,1	2,9	0,40	0,14
		Rokanol Ł-18	1,3	3,1	0,46	0,15
Pseudoplasticity coefficient n	BO Green	Rokanol L-10	0,08	0,26	0,12	0,27
		Rokanol Ł-18	0,08	0,28	0,17	0,33
	RLL Red	Rokanol L-10	0,06	0,21	0,11	0,14
		Rokanol Ł-18	0,07	0,24	0,14	0,18
Activation energy of viscous flow E_η kJ/mol	BO Green	Rokanol L-10	4,9	29,0	3,2	4,65
		Rokanol Ł-18	5,3	30,0	3,7	4,70
	RLL Red	Rokanol L-10	2,8	28,0	2,3	4,30
		Rokanol Ł-18	3,3	28,5	2,8	4,40

Table 5

Constant values for equations (9) – (11) describing changes in maximum viscosity, pseudoplasticity coefficient and activation energy of viscous flow for plastisol containing a fixed surfactant and thickener contents and variable concentrations of organic pigments and water

Rheological parameter	Pigment type	Surfactant type	Constant type			
			a*10	b	c*10	d
Maximum viscosity $\lg \eta_0$ [mPas]	BO Green	Rokanol L-10	2,6	0,70	0,9	3,75
		Rokanol Ł-18	2,8	0,95	1,1	3,9
	RLL Red	Rokanol L-10	1,6	0,45	0,5	3,5
		Rokanol Ł-18	2,1	0,60	0,7	3,6
Pseudoplasticity coefficient n	BO Green	Rokanol L-10	0,41	0,25	0,18	0,20
		Rokanol Ł-18	0,45	0,30	0,19	0,22
	Czerwień RLL	Rokanol L-10	0,24	0,15	0,10	0,16
		Rokanol Ł-18	0,33	0,20	0,13	0,18
Activation energy of viscous flow E_η [kJ/mol]	RLL Red	Rokanol L-10	34,1	22,0	11,4	30,0
		Rokanol Ł-18	41,0	24,0	13,8	31,0
	RLL Red	Rokanol L-10	19,5	18,0	4,9	28,0
		Rokanol Ł-18	26,8	20,0	6,5	29,0

a increased plastisol viscosity. It turns out it increases in correlation with the increasing amount of water in the plastisol. The data shown it Table 5 points directly to such a mechanism of thickener interaction in the plastisol, which in case of maximum viscosity and viscous flow activation energy are multiple times larger than those presented in Table 4. In both tables the values describing changes in pseudoplasticity coefficient are similar. This suggests, that the mechanism of bond network destruction and water micelle (containing the surfactant and thickener) deformation due to shear stress is similar.

Summary

Rheological analysis of PVC plastisols containing a fixed amount of surfactant and increasing water content shows, that around water content value of 30.0% weight in plastisol, a phase inversion phenomenon takes place. Essentially the plastisol, which was thus far the dispersive phase, became the dispersed phase in water, stabilised by adsorption layers of a surfactant. Subsequent studies were concerned with introducing a plastisol containing a constant amount of non-ionic surfactant with variable amounts of organic pigment and water. These in turn show, that in such compositions the increase in organic pigment content results in increasing viscosity, while increasing the amount of water results in its decrease. Afterwards a plastisol containing a constant amount of non-ionic surfactant was introduced with a fixed amount of polyurethane thickener and increasing amounts of both water and organic pigment. In all cases the increasing amounts of organic pigment and water resulted in increasing viscosity of the plastisol composition. It was shown, that viscosity changes are more significant, the larger specific surface area of the pigment and the ethoxylation number of the non-ionic surfactant.

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