Effect of PVC plastisol composition and processing conditions on foam expansion and tear strength

Rajko Radovanović[1], Vladislav Jašo[2], Branka Pilić[2], Dragoslav Stoiljković[2]

[1]*OOO JUTEKS RU, Kameshkovo, Russia*
[2]*Faculty of Technology, University of Novi Sad, Novi Sad, Serbia*

Abstract

The quality of vinyl floorings depends mainly on the right control of the formation of poly (vinyl chloride) (PVC) foam structure. Many process parameters influence the cellular structure and final properties of the PVC foam. In this paper the influence of concentration of blowing agent and calcium carbonate filler, as well as temperature and time of the blowing process on the expansion ratio and tear strength of the PVC foam, were studied. Moreover, regression analysis was performed in order to determine the significance of studied parameters influence on expansion ratio and tear strength of PVC foams. It was found that concentration of the blowing agent in the plastisol mixture had the principal influence on the expansion ratio of the PVC foam. Tear strength was found to depend more or less equally on all studied parameters. The study has also shown that the addition of calcium carbonate filler had insignificantly lowered the expansion ratio, but at the same time it could significantly lower the cost of the final product. This effect was practically employed to improve the economic efficiency of the PVC floorings production in JUTEKS plant in Russia.

Keywords: PVC plastisol; foam expansion, tear strength.

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In the last decade of the previous century demand for polymeric foams has risen dramatically [1]. The growth in demand has been more dramatic in the United States than in Europe. In recent years, this trend of growth has continued at a rapid pace in the whole world. The cause of this are the numerous excellent properties that encompass light weight, excellent strength/weight ratio, superior insulating abilities, and energy absorbing performance and comfort features of polymeric foams [2]. The application possibilities of polymeric foams are very wide and include furniture, transportation, bedding, carpet underlay, packaging, textiles, toys, gasket, sport applications, and insulation appliances.

Early investigations about PVC foams dealt with the effect of viscosity regulators and how they can be used to control the process of foam formation [3]. Later, studies were concerned with how tensile and flexural properties, as well as toughness correlated to the density of foamed PVC [1]. It was found that as density decreases, so do all the mentioned properties [1]. Foam properties are also found to be dependent on and influenced by the cell structure [4]. A study which encompassed analysis of four different PVC types and eight different plasticizers, individually or as mixtures, on foam density, ratio of expansion and elasticity [5] showed that two-component plasticizer mixtures had better properties than single-component plasticizers. This study has shown that most appropriate mixtures are those of di-isoheptyl phthalate and butylbenzyl phthalate [5]. It was also shown that the precise timing of foam expansion and careful choice of temperature are critical for the foam properties. If the temperature is too high, the degradation of PVC becomes very rapid and decreases the quality of the product. At high temperatures, the melt viscosity is low so rapid gas expansion can cause cell rupture and collapse [6]. This results in non-uniform cell morphology, increased foam density and increased roughness. On the other hand, if the temperature is too low, decomposition of the blowing agent is too slow, which can considerably influence the productivity. Moreover, at low temperatures fusion between the PVC primary particles is insufficient. The plastisol is characterized by high viscosity in lower temperature range, so if the gas pressure is not high enough it may cause a decrease in cell size, increase in foam density, surface roughness and void formation.

Different studies have dealt with the influence of the stabilizer type [7], type and level of the acrylic processing aid [8], and processing conditions [9] on foam density and thus on the foam properties. Heat stabilizers such as Ca/Zn stearates or organotin compounds are added to the PVC to prevent dehydrochlorination by heating. The heat stabilizers also affect the rate of decomposition of chemical blowing agents [10,11].

Correspondence: R. Radovanović, OOO JUTEKS RU, Dorozhnaya Street 10, 601301 Kameshkovo, Russia.
E-mail: Rajko.Radovanovic@juteks.ru
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The effect of the $CaCO_3$ particle size was studied in rigid PVC formulations [12–14]. Study of particle size effect in foam formulations [15] proved that smaller particle size enables efficient fusion of the compound. However, larger particle size fillers improve melt strength, giving foam with more homogeneous cell size distribution and smaller fraction of open cells. In that way, mechanical properties are improved [15]. It was observed that, samples containing zeolite, $CaCO_3$, cellulose or luffa flour had lower pore volume, but higher Young's modulus and stress values compared to unfilled samples [2]. Finally, at low filler concentration, filler acts as nucleating agent and increases the foam formation. The structure of PVC foams becomes rigid and strong at high filler concentration. It is observed that the properties of cellulose or luffa added foam are not suitable for practical foam applications such as thermal insulation and construction field, but can be used in plant watering equipment since they have high water uptake capacity [2].

In this study, we have investigated the effect of four process parameters on expansion ratio and tear strength of PVC foam. The parameters that were studied are temperature (in the stabile range of 180–196 °C), expansion time (in the range 90–150 s), calcium carbonate filler concentration, and azodicarbonamide blowing agent concentration. Multiple regression analysis was employed in order to determine the significance of the influence of the different parameters on the expansion ratio and tear strength.

EXPERIMENTAL

The plastisol consists of PVC powder (Solvic 367SF, Solvin, France), PVC extender (Solvic 266SF, Solvin, Belgium), dioctyl phthalate plasticizer (Roshalskiy Zavod Plastifikatorov, Roshaly, Russian Federation), azodicarbonamide blowing agent (MC-2, OOO Norteks, Moscow, Russian Federation), ZnO kicker (BC OM, Bellit, Saransk, Russian Federation), TiO_2 (R103, DuPont, Germany), calcium carbonate filler (Omycarb 40 UR, Omya Ural, Russian Federation, with particle size 26±5 μm), viscosity regulator (Viskobyk 4041, BYK Chemie, Germany) and dispersing agent (Dispersplast 1138, BYK Chemie Germany). Nine different formulations were prepared with three different calcium carbonate concentrations (40, 70 and 100 phr) and three different blowing agent concentrations (0.8, 1 and 1.2 wt.%). The ratio of blowing agent to kicker percent was 3:1 in all formulations. In total, there were nine different formulations (Table 1). One formulation was shown as an example in Table 2.

Table 2. PVC plastisol formulation (sample M1)

Ingredient	Concentration	
	phr	wt.%
PVC, Solvic 367NF	75.52	37.04
Ext. PVC, Solvic 266SF	24.48	12.00
DOP	55.94	27.44
$CaCO_3$	40.17	19.70
DOP (batch)	3.24	1.59
Viskobyk 4041	1.55	0.76
ADCA azodicarbonamide	1.64	0.80
ZnO	0.55	0.27
TiO_2	0.40	0.19
Disperplast 1138	0.40	0.19
Total	203.9	100.0

Poly (vinyl chloride) plastisols were prepared by mixing the appropriate components in a laboratory vacuum mixer (LPE, Werner Mathis AG, Switzerland). The prepared plastisols were coated on a silicon paper and were left to gel for 30 s at 150 °C in an oven (LTF, Werner Mathis AG, Switzerland). The samples were expanded into foam at five different temperatures (180, 184, 188, 192 and 196 °C) and in three different time periods (90, 120 and 150 s). The expansion of PVC foam was measured by comparing the height of the sample with the height of the control sample that was processed at 150 °C for 30 s and expressed as expansion ratio that shows how many times thickness has changed after expansion. The tear strength was measured by mechanical test machine (Zwick/Roell Z005) on samples 50 mm wide and 200 mm long with a longitudinal 100 mm slit along the middle at a tearing rate of 100 mm/min.

RESULTS

The main goal of this study was to determine which of the four studied parameters (concentration of calcium carbonate, concentration of blowing agent, tem-

Table 1. Plastisol samples designation and concentration of azodicarbamide blowing agent (ADCA), ZnO kicker and calcium carbonate filler (wt.%)

Ingredient	Sample								
	M1	M2	M3	M4	M5	M6	M7	M8	M9
ADCA	0.8	0.8	0.8	1.0	1.0	1.0	1.2	1.2	1.2
ZnO	0.27	0.27	0.27	0.33	0.33	0.33	0.4	0.4	0.4
$CaCO_3$	19.70	29.87	37.58	19.51	29.57	37.22	19.32	29.28	36.86

perature and foam expansion time) has the most significant influence on the expansion ratio and tear strength of the foam. Efforts were made to find the appropriate linear correlation with the least number of independent variables that would predict both of the mentioned properties with an acceptable level of accuracy. It is well known that with the increase of temperature, the rate of blowing agent decomposition increases thus allowing the greater expansion ratio. It should also rise with the time allowed for the foam formation, as well as with the higher concentration of blowing agent. Special attention was given to the influence of the calcium carbonate in the plastisol on the expansion ratio. The parameters that were studied were in the range used under normal production conditions, only the calcium carbonate percent was increased above the usual level. Temperatures were above the temperature which enables satisfactory rate of blowing gas evolution and beneath the level the degradation of PVC becomes considerable.

After the preparation of samples and measurements of foam properties, multiple linear regression was performed in order to study the effect of different parameters on the expansion ratio and tear strength. In the first step, linear correlation (1) between expansion ratio and each parameter was separately determined (Table 3):

$$E = a_i X_i, \quad i = 1,...,4 \tag{1}$$

The highest value of correlation coefficient (*R*) and the lowest root mean square error (*RMSE*) among four correlations indicates that azodicarbonamide blowing agent concentration (ADCA) has the biggest effect on the expansion ratio of PVC foam by far.

Table 3. Correlation factor (R) and root mean square error (RMSE) of correlations between expansion ratio and each single studied variables (Model E1)

Parameter	Variable designation	*R*	*RMSE*
ADCA, wt.%	*X*2	0.774	0.5041
Temperature, °C	*X*4	0.420	0.7226
$CaCO_3$, wt.%	*X*1	0.181	0.7831
Time, s	*X*3	0.236	0.7737

Further on, forward stepwise analysis was performed, introducing new parameters in the model in the order of their significance. Models are given below:

$$E = a_2 X_2 \tag{E1}$$

$$E = a_0 + a_2 X_2 + a_4 X_4 \tag{E2}$$

$$E = a_0 + a_2 X_2 + a_4 X_4 + a_3 X_3 \tag{E3}$$

$$E = a_0 + a_2 X_2 + a_4 X_4 + a_3 X_3 + a_1 X_1 \tag{E4}$$

The results of step two (two parameter model) and step three (three parameter model) are shown in Tables 4 and 5. First, we have tried to improve model E1 by including one more parameter. Effect of inclusion of each of the three remaining parameter was explored. The values of parameter coefficients (a_i), reduction in error sum of squares (*SS*), if the term is entered into the model and F ratio is given in the tables. *F* ratio is defined as the ratio of the explained variance to the unexplained variance and expresses the significance level of each parameter in F statistic. In our case it shows what would be the significance of the particular parameter in the model if it was included in the model. Higher value means higher significance.

Table 4. Model E2 coefficients reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	–10.3979	0	0
$CaCO_3$, wt.%	0	3.56	30.43
ADCA, wt.%	3.7464	50.52	352.54
Time, s	0	4.70	43.37
Temperature, °C	0.0586	14.87	103.79

Table 5. Model E3 coefficients, reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	-11.7544	0	0
$CaCO_3$, wt.%	0	3.56	43.54
ADCA, wt.%	3.7464	50.52	465.71
Time, s	0.0076	4.70	43.37
Temperature, °C	0.0586	14.87	137.11

If we look at correlation coefficients (*R*) and root mean square error (*RSME*, Table 6) for different correlations, we can see that introduction of the third and fourth term does not give much of an improvement. Because Model E2, with two parameters, is simpler, it was our model of choice. Comparison of the values of the expansion ratio predicted by the model and the experimental values were compared and the points that differed more than 2σ were disbanded (6 points from the total of 135 points) and calculation of the new improved model coefficients was performed (Table 7). All three models are shown in Figures 1 and 2.

Although analysis has indicated that concentration of the blowing agent (ADCA) is the most important factor that determines the expansion ratio, the processing parameters are very important as well. This is illustrated in Figures 3 and 4. The obtained results show

that foaming of PVC can be performed in a very narrow range of temperature and time. It is clear that foaming below 180 °C is difficult and insufficient. This can be explained by the fact that below 175 °C decay of the blowing agent does not take place and foaming does not start no matter how long the PVC plastisol is heated. On the other hand, at higher temperatures, longer time of heating does not give foam with much higher expansion ratio. Also worth mentioning is that above the temperature of 195 °C and 150 s, the degradation of the PVC starts and intensifies after reaching 200 °C and longer foaming times. Optimal processing conditions are in temperature range of 188–192 °C with a foaming time of 120 s.

Table 6. Correlation factor (R) and root mean square error (RMSE) for different correlation models for expansion ratio dependence on studied parameters

Model	Model parameters	*R*	*RMSE*
E2	*X*2, *X*4	0.881	0.3786
E3	*X*2, *X*4, *X*3	0.912	0.3294
E4	*X*2, *X*4, *X*3, *X*1	0.935	0.2862

Table 7. Model E2I coefficients, reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	–8.4382	0	0
ADCA, %	3.8195	50.75	544.39
Temperature, °C	0.0458	8.21	88.12

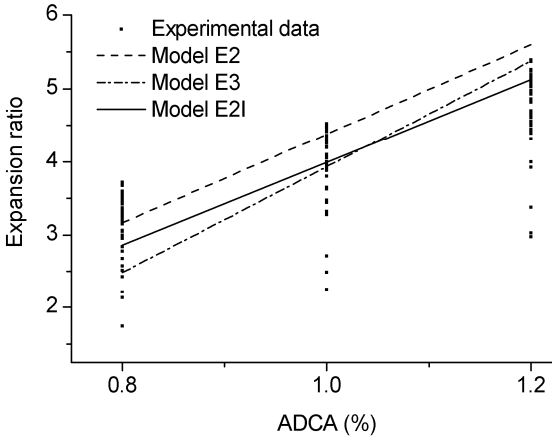

Figure 1. Experimental data of expansion ratio for different concentrations of blowing agent and different correlation models.

Adding calcium carbonate filler has negative effect on the expansion ratio, but significance of its effect (expressed as *F* ratio) is relatively low (Tables 3 and 4). This information is very important as it shows that with the addition of the filler, the price of the product can be lowered with an insignificant change of the expansion ratio.

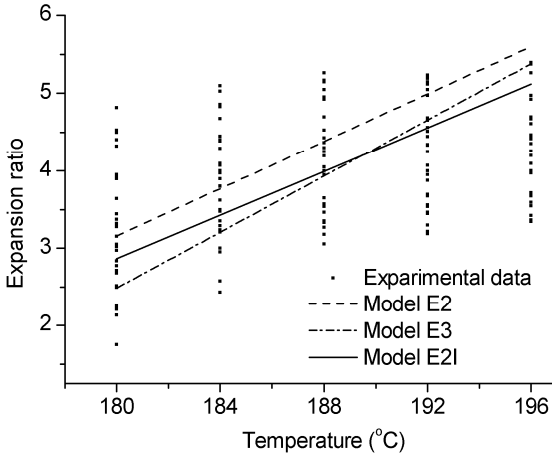

Figure 2. Experimental data of expansion ratio for different temperatures and different correlation models.

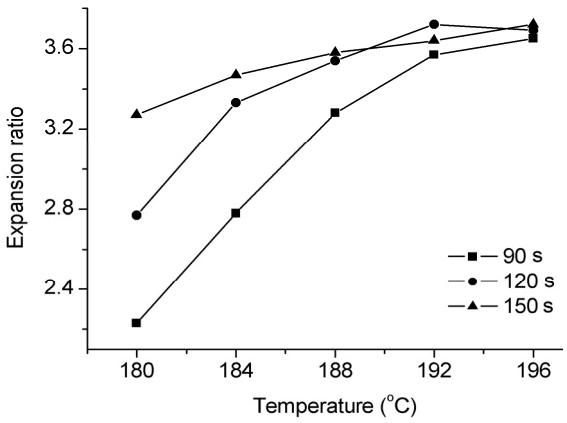

Figure 3. Experimentally obtained dependences of expansion ratio on temperature for different foaming time (Sample M3).

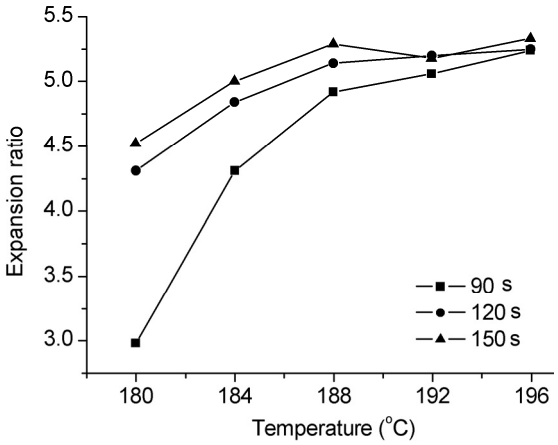

Figure 4. Experimentally obtained dependences of expansion ratio on temperature for different foaming time (Sample M9).

The effect of the studied factors on tear strength was also examined by statistical analysis. Correlation models are given below.

$$T = a_2 X_2 \quad \text{(T1)}$$

$$T = a_0 + a_2 X_2 + a_4 X_4 \quad \text{(T2)}$$

$$T = a_0 + a_2 X_2 + a_4 X_4 + a_3 X_3 \quad \text{(T3)}$$

$$T = a_0 + a_2 X_2 + a_4 X_4 + a_3 X_3 + a_1 X_1 \quad \text{(T4)}$$

Separate correlation between tear strength and each parameter was determined (Table 8). Again, concentration of the blowing agent (ADCA) and temperature are the most significant factors, but the calcium carbonate concentration also has a significant effect. The stepwise analysis routine was performed to find model coefficients (Tables 9 and 10). In the case of the model describing the effects of the studied factors on tear strength, it was decided that model with three parameters (Model T3) is more accurate than the model with two parameters (Model T2, Table 11). This model was then improved by recalculation of the model coefficients after the disbandment of experimental points outside the 2 σ region (Table 12).

Table 8. Correlation factor (R) and root mean square error (RMSE) for the correlations of tear strength and each single studied variables (Model T1)

Parameter name	Variable designation	*R*	*RMSE*
$CaCO_3$, wt.%	*X*1	0.421	2.925
Temperature, °C	*X*4	0.458	2.866
ADCA, %	*X*2	0.468	2.849
Time, s	*X*3	0.233	3.135

Table 9. Model T2 coefficients, reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	63.0125	0	0
$CaCO_3$, wt.%	0	262.02	65.04
ADCA, wt.%	–9.1645	302.36	50.53
Time, s	0	74.70	13.68
Temperature, °C	–0.2591	290.01	48.47

From the above analysis it may be concluded that all factors have negative effect on tear strength. This is understandable as the cellular structure weakens the strength of material to resist tearing. The tear strength decreases with increase of concentration of the calcium carbonate in the foam (Figures 5 and 6). Decrease of the tear strength is less dramatic if there is a higher concentration of blowing agent. At higher temperatures (Figure 6) all other factors have much less influence as the tear strength is low and could not be much increased by changing other factors.

Table 10. Model T3 coefficients, reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	68.8031	0	0
$CaCO_3$, wt.%	–0.1920	260.02	65.04
ADCA, wt.%	–9.4317	319.93	79.42
Time, s	0	74.70	21.44
Temperature, °C	–0.2591	290.01	71.99

Table 11. Correlation factor (R) and root mean square error (RMSE) for different correlation models for tear strength dependence on studied parameters

Model name	Model parameters	*R*	*RMSE*
Model T2	*X*2, *X*4	0.655	2.446
Model T3	*X*2, *X*4, *X*1	0.786	2.007
Model T4	*X*2, *X*4, *X*1, *X*3	0.819	1.867

Table 12. Model T3I coefficients, reduction in sum of squares and F ratio after introduction of different parameters to the model (from Analysis of variance)

Parameter	Model coefficient	Sum of squares	*F* Ratio
Intercept	50.6867	0	0
ADCA, wt.%	–7.7716	209.08	210.91
Temperature, °C	–0.1771	124.91	125.99
$CaCO_3$, wt.%	–0.1649	183.85	185.45

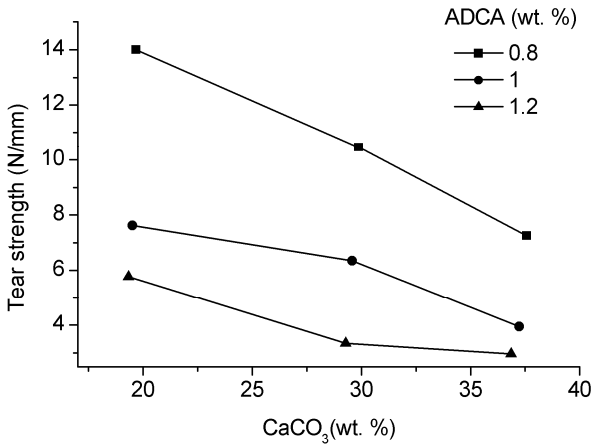

Figure 5. The dependence of tear strength on calcium carbonate concentrations for different blowing agent concentrations (184 °C, 90 s).

The correlation between experimental data of the tear strength and expansion ratio was also tested. This analysis (Figure 7) has shown that some correlation between the two properties exists although it is not

significant. This could be explained by the fact that tear strength depends only partially on expansion ratio or density of the foam, but more importantly on the cell structure and dimension of pores. This dependence will be studied in our further investigations.

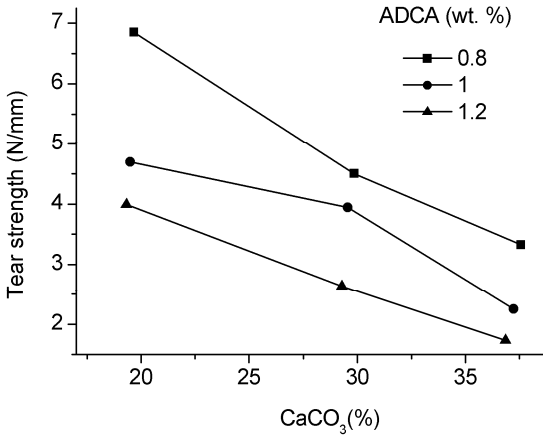

Figure 6. The dependence of tear strength on calcium carbonate concentrations for different blowing agent concentrations (196 ℃, 90 s).

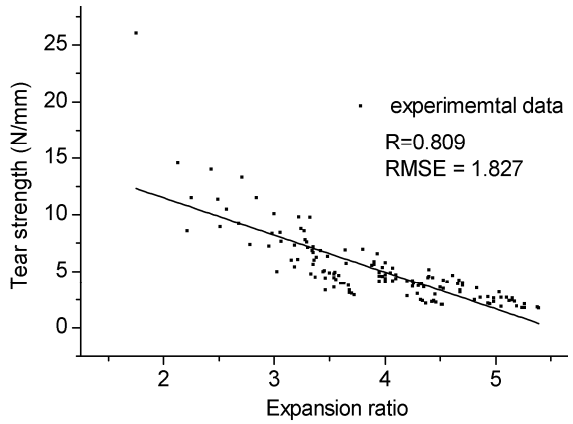

Figure 7. Correlation between tear strength and expansion ratio of the PVC foam (all 135 specimens: formulation M1–M9; time: 90, 120 and 150 sec; temperature: 180, 184, 188, 192 and 196 ℃).

CONCLUSIONS

In this study the effect of temperature, foaming time, calcium carbonate and blowing agent concentration on expansion ratio and tear strength was investigated. Statistical analysis was performed in order to study the significance of the influence of different factors on the expansion ratio and tear strength. Correlation models that describe these relations were developed. It was found that the expansion ratio of PVC foam is mostly dependent on the concentration of blowing agent and temperature. Both factors have a positive influence on the expansion ratio. Increase of the calcium carbonate concentration in the foam lowers the expansion ratio, but its influence was found to be the least significant among the four studied parameters. These findings confirm usefulness of calcium carbonate to lower the cost of the PVC foam products. In contrast to the influence of the expansion ratio, the increase of the concentration of calcium carbonate and ADCA, as well as temperature, has a negative impact on the tear strength. This means that the mixture formulation and the process conditions must be optimized in order to obtain the desired characteristics and sustainable cost.

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IZVOD

UTICAJ SASTAVA PVC PLASTISOLA I USLOVA PROIZVODNJE NA STEPEN EKSPANZIJE PENA I OTPORNOST NA CEPANJE

Rajko Radovanović[1], Vladislav Jašo[2], Branka Pilić[2], Dragoslav Stoiljković[2]

[1]OOO JUTEKS RU, Dorozhnaya street 10, 601301 Kameshkovo, Russia
[2]Tehnološki fakultet, Univerzitet u Novom Sadu, Novi Sad, Srbija

(Naučni rad)

Kvalitet PVC podova zavisi pre svega od kontrole nastanka ćelijske strukture polivinilhlorida (PVC) pena. Ćelijska struktura i krajnja svojstva PVC pene zavise od mnoštva proizvodnih prametara. U ovom radu izučavan je uticaj koncentracije sredstva za penjenje i kalcijum karbonata kao punila, kao i temperature i vremena penjenja na stepen ekspanzije i otpornosti na cepanje PVC pena. Osim toga, urađena je regresiona analiza u cilju utvrđivanja značaja navedenih parametara na stepen ekspanzije i otpornost na cepanja PVC pena. Utvrđeno je da koncentracija sredstva za penjenje u smeši za penjenje ima presudan uticaj na stepen ekspanzije PVC pena. Otpornost na cepanje, pokazalo se, zavisi podjednako od svih izučavanih parametara. Pokazano je da dodatak kalcijum karbonata kao punioca ima vrlo mali uticaj na smanjenje stepena ekspanzije ali zato, sa druge strane, značajno utiče na smanjenje cene krajnjeg proizvoda. Ovo je primenjeno u praksi radi povećanja ekonomske efikasnosti proizvodnje PVC podova u fabrici JUTEKS u Rusiji.

Ključne reči: PVC plastisol • Ekspanzija pena • Otpornost na cepanje