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KOMPOZYTY
COMPOSITES

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RHEOLOGY, EXTRUDABILITY AND MECHANICAL PROPERTIES OF CERAMIZABLE SILICONE COMPOSITES

Ceramizable silicone composites of various origin, designated as A, B and C, which can be used for insulation jackets of electrical cables, were the subject of investigations. The rheology and processing studies of the materials were carried out with a one-screw laboratory Brabender Measuring Extruder 19/10 DW (Germany), operating with a screw rotational speed from 10 to 200 rpm. For evaluation of the rheological characteristics of the mixes, $\eta = f(\gamma_R)$, an oval capillary die head was used, whereas a Garvey's head was mounted to the machine for determination of the extrusion rate, linear shrinkage and swelling of the extrudate (Barrus' effect). Extrudability studies of the mixes were carried out according to ASTM D 2230-83. The vulcametric characteristics of the materials contained 2,4 (di)chloro benzoyl peroxide, as a curing agent, were evaluated with a Monsanto 100 (USA) vulcameter, according to ISO 3417. The mechanical properties - tensile strength (TS) and tear strength (TES) of the crosslinked composites were determined with a universal mechanical testing machine Zwick 1435 (Germany) according to ISO 37 and ISO 34 standards respectively. Despite similar rheological characteristics, mix A exhibits the best extrudability. It manifests itself by the highest extrusion rate, minimum linear shrinkage and the lowest expansion of the extrudate among the materials studied. All the mixes studied can be extruded with a wide range of screw rotational speed. The higher the rotational speed, the better the efficiency of extrusion, which meets industrial expectations. Edge defects appear on the surface of the extrudates when the rotational speed is low (below 60÷90 rpm). The mechanical properties of the composites studied meet the requirements of the cable industry: *TS* = 7÷9 MPa, *TES* = 12÷15 kN/m and $E_B \approx 300\%$. The composition of the mineral phase, in a glaze and silicone rubber matrix, is responsible for the differences in the extrudability of the composites studied. Composites containing small particles of milled quartz and wollastonite dominate over the one based on large particles of mica and zinc oxide, regarding the processing parameters.

Keywords: **cables, ceramizable silicone composites, rheological characteristic, extrudability, mechanical properties**

REOLOGIA, WYTŁACZALNOŚĆ I WŁAŚCIWOŚCI MECHANICZNE SILIKONOWYCH KOMPOZYTÓW CERAMIZUJĄCYCH

Obiekt badań stanowiły silikonowe kompozyty ceramizujące, oznaczone jako A, B i C, które mogą być stosowane na osłony przewodów elektrycznych. Badania reologiczne i próby wytłaczalności materiałów wykonano z wykorzystaniem jednoślimakowej wytłaczarki laboratoryjnej Measuring Extruder 19/10 DW firmy Brabender (Niemcy). Do wyznaczenia charakterystyki reologicznej mieszanek $\eta = f(\gamma_R)$ zastosowano głowicę kapilarną o owalnym przekroju, podczas gdy do oznaczania parametrów wytłaczania: szybkości wytłaczania, skurczu liniowego i pęcznienia (efektu Barrusa) wytłoczyny, do wytłaczarki zamontowano głowicę z formownikiem Garveya. Wytłaczalność mieszanek oceniano według ASTM D 2230-83. Charakterystyki wulkametryczne materiałów, zawierających jako środek sieciujący nadtlenek 2,4 (di)chloro benzoilu, wyznaczano za pomocą wulkametru Monsanto 100 (USA), według ISO 3417. Właściwości mechaniczne usieciowanych kompozytów: wytrzymałość przy zerwaniu (TS) oraz odporność na rozdzieranie (TES) oznaczono za pomocą uniwersalnej maszyny wytrzymałościowej Zwick 1435 (Niemcy), odpowiednio według norm ISO 37 i ISO 34. Pomimo zbliżonych charakterystyk reologicznych mieszanka A wytłacza się najlepiej. Przejawia się to w zdecydowanie największej wartości szybkości wytłaczania, minimalnym skurczu liniowym i najmniejszym pęcznieniu wytłoczyny spośród badanych materiałów. Wszystkie badane mieszanki można wytłaczać w szerokim zakresie prędkości obrotowej ślimaka. Im większa szybkość obrotowa, tym lepsza efektywność procesu, co jest zgodne z oczekiwaniami przemysłu. Defekty krawędziowe pojawiają się na powierzchni wytłoczyn przy małych prędkościach obrotowych ślimaka (poniżej 60÷90 obr./min). Właściwości mechaniczne badanych kompozytów spełniają wymagania stawiane przez przemysł kablowy: *TS* = 7÷9 MPa, *TES* = 12÷15 kN/m i $E_B \approx 300\%$. Skład fazy mineralnej, oprócz szkliwa i matrycy z kauczuku silikonowego, jest odpowiedzialny za wytłaczalność badanych kompozytów. Kompozyty zawierające małe cząstki mielonego kwarcu i wolastonitu mają zdecydowaną przewagę nad materiałami bazującymi na dużych cząstkach miki i tlenku cynku, biorąc pod uwagę parametry przetwórstwa.

Słowa kluczowe: **kable, silikonowe kompozyty ceramizujące, charakterystyka reologiczna, wytłaczalność, właściwości mechaniczne**

INTRODUCTION

Despite significant progress in the area of fire protection and fire fighting, building fires still remain dangerous for human life and result in considerable material losses. The methods and conditions of fire protection for buildings are regulated by Polish law [1-3]. Strict regulations concern objects of special function like schools and other buildings serving educational purposes, trade and recreation centers, supermarkets, hospitals, sport halls, airports, multi-storey buildings, cinemas, theatres, museums, art galleries, railways and metro stations, briefly speaking, all those places where a great number of people are usually present or goods of significant cultural and/or material value have been collected. According to the existing regulations, the functioning of monitoring systems, fire signalization and power supply installations have to be guaranteed for 90 min. Electrical cables used in such installations are assigned PH 90, according to PN-EN-50200 and PN-IEC 60331-31:2004 standards. The standards describe the methodology of checking electrical cables for both flame resistance and electrical integrity of the circuits during fire.

Silicone rubber, chlorosulphonated polyethylene, poly(vinyl chloride) or crosslinked polyethylene, still used till not so long ago by the cable industry, are not able to meet the present requirements, mainly because of their low mechanical strength at high temperatures arising during fire, which can even reach the melting temperature of copper (1083°C), and the limited thermal stability of polymers. A solution to the problem showed to be the application of composites based on silicone rubber, but filled with a ceramic phase containing silicates, metal oxides and hydroxides, their salts and other chemical compounds, facilitating the production of concise and strong coating in a wide range of temperature from 450°C (degradation of silicone rubber) up to 1050°C (close to the melting temperature of copper, being the base material for wires) [4, 5]. There are numerous patents where the optimization of a ceramic phase composition has been made [6-9].

Silicone ceramizable composites are very high loaded systems, of filler content at the level of 100-120 phr. The characteristic of the filler particles decides the mechanical properties of the composites and it is important from the point of view of mix preparation and its further processing by extrusion. There are two problems associated with this fact. The first one concerns the mechanical requirements: *TS* = 7÷9 MPa and TES = = 12÷15 N/mm, demanded by the cable industry. The second one deals with the influence of silicone rubber, the composition and content of the solid phase, as well as the conditions of their mixing, on the rheological characteristics and extrudability (technology used for cable production) of the composites. In the work, the rheology, extrusion parameters, extrudability, vulcametric characteristics and mechanical properties of some commercially available silicone ceramizable composites used by the cable industry have been determined and compared.

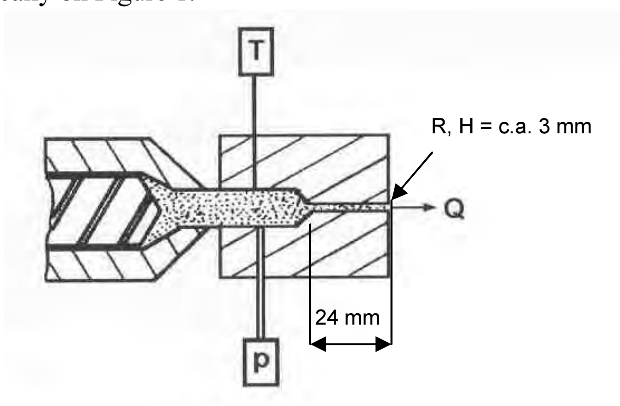
MATERIALS AND METHODS

Three silicone ceramizable composites used for the insulation of electrical cables of various origin, A, B and C, were the subject of the studies. The composition and ceramization of the materials were analyzed in our previous work [10]. Detailed information on the composition of the silicone composites studied is confidential. They differ significantly according to the glaze and mineral phase being used. The composition of their mineral phase has been compared in Table 1.

TABLE 1. **Mineral phase composition of silicone composites studied**
TABELA 1. **Skład fazy mineralnej badanych kompozytów silikonowych**

Silicone composite	Mineral phase composition [wt. %]				
	Wollastonite $CaSiO_3$	Quartz SiO_2	Kaolinite	ZnO	Mica (Muscovite)
A	52.0	40.2	7.8	-	-
B	-	96.5	3.5	-	-
C	-	-	-	35.5	64.5

This time, the rheology, extrusion parameters, extrudability, vulcametric characteristics and mechanical properties of the composites, crosslinked with 2,4 (di)chloro benzoyl peroxide, were compared. The rheological studies were carried out with a one-screw laboratory Brabender Measuring Extruder 19/10 DW (Germany) of the basic characteristics: screw diameter *D* = = 19 mm; screw length *L* = 190 mm (*L/D* = 10); number of heating zones in a cylinder: 1; max. torque 150 Nm; max. temperature of operation 300°C; max. mass pressure 700 bar; processing ability 0.5÷5 kg/hr, equipped with an oval capillary die head, demonstrated schematically on Figure 1.



T, P - temperature and mass pressure sensors respectively

Fig. 1. Scheme of oval capillary die head, used in investigations
Rys. 1. Schemat głowicy kapilarnej o owalnym przekroju wykorzystanej w badaniach

The experiment was based on extrusion of the materials studied with a different screw rotational speed in the range of 5 to 140 rpm. For any particular speed, three determinations were performed and the following rheological parameters averaged (1)-(3):

- shear stress $\tau = \frac{\Delta p \cdot H}{2L}$ [Pa] (1)

- shearing speed $\gamma = \frac{6Q}{\pi R^3}$ [s^{-1}] (2)

- kinematic viscosity $\eta = \frac{\tau}{\gamma}$ [Pa·s] (3)

where: Δp - gradient of pressure [Pa]; R, L, H - radius, length and height of the capillary respectively [cm]; Q - volumetric rate of flow [cm^3/s].

Polymer materials are non-Newtonian liquids, so to calculate the real value of shearing speed (γ_R), the correction proposed by Rabinovitsch was applied:

$$\gamma_R = \frac{\gamma}{4} \cdot \left(3 + \frac{d \cdot \log \gamma}{d \cdot \log \tau} \right) \quad (4)$$

where d - material density [g/cm^3].

Based on the experimental data and performed calculations, the rheological characteristics, $\gamma_R = f(\tau)$, for the mixes studied were prepared. Using the same base extruder unit, this time with a Garvey's die head, the following processing parameters were determined:

- extrusion rate $V_w = l_{15} / t$ [cm/min] (5)
  where: l_{15} - length of extrudate after 15 s
  t - time of extrusion, t = 15 s
- linear shrinkage of extrudate, measured after 30 min
  $S_L = 100\,(l_{15} - l_{30}) / l_{15}$ (6)
  where: l_{15} - length of extrudate after 15 s; l_{30} - length of extrudate after 15 s, measured after 30 min,
- swelling (W_p) - the so-called Barrus' effect [6]
  $W_p = 100\,((g / d \cdot l_{15} \cdot A) - 1)$ (7)
  where: g - mass of extrudate after 15 s [g]; d - material density [g/cm^3]; A - area of channel die (0.47 cm^2).

Quantitative analysis was carried out at the temperature of 50 and 60°C, for the head and extruding die respectively, applying the rotational screw speed of: 10, 30 and 50 rpm.

The extrudability of the composites was evaluated using the same head, under the same temperature conditions, but at a higher rotational screw speed, from 60 up to 200 rpm, according to the ASTM D 2230-83 standard. Qualitative analysis was applied to the edges and surface of the extrudates, from the point of view of their porosity and smoothness. During the experiments, the temperature, mass pressure in the cylinder and screw rotational speed were monitored simultaneously.

The vulcametric characteristics of the materials containing 1.8 phr of 2.4 (di)chloro benzoyl peroxide, were evaluated with a Monsanto 100 (USA) vulcameter at 120°C, according to ISO 3417. The mechanical properties of the crosslinked composites: tensile strength (TS) and tear strength (TES), were determined with a mechanical testing machine, Zwick 1435 (Germany), according to ISO 37 and ISO 34 standards respectively.

RESULTS AND DISCUSSION

The rheological characteristics of the mixes studied are compared in Figure 2.

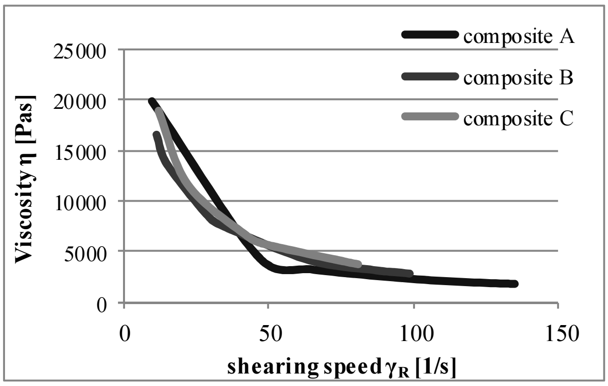


Fig. 2. Rheological characteristics $\eta = f(\gamma_R)$ of the silicone mixes studied

Rys. 2. Charakterystyki lepkościowe $\eta = f(\gamma_R)$ badanych mieszanek silikonowych

The above data indicate a slightly lower viscosity of mix A in comparison to the other materials studied, especially at a higher shearing speed. At a lower shearing speed, the situation is reversed. However, one should keep in mind that the extrusion of cable jackets takes place at very high rates. The materials are resistant to ageing. Their rheological characteristics have slightly deteriorated after one year of storage, which was followed by worse parameters of extrusion. Nevertheless, the values of the extrusion rate, linear shrinkage or swelling did not change much. For example, of mix A, the first parameter decreased by c.a. 7%, the second one increased by 0.8% and the third one decreased by 9%. The material still remains useful for extrusion.

The parameters of the extrusion process for the materials studied, at three different screw rotational speeds, are compared in Table 2.

The extrusion rate of the mixes studied is the smallest at low screw rotational speed, but increases significantly (almost monotonously) with an increasing rotational speed. In this case, improvement of the extrudability is accompanied by a decreasing linear shrinkage, S_L, but unfortunately an increase in swelling W_p. Mix (A) exhibits the highest extrusion rate and the lowest Barrus' effect among the materials studied, being practically free from linear shrinkage. Mixes B and C are different from the A one. Despite similar rheological characteristics, the first and especially the second material exhibit a significantly lower extrusion rate, especially at the highest screw rotational speed, preferred by the cable industry. The difference is over 50% at a screw rotational speed of 50 rpm in comparison to mix A.

TABLE 2. **Extrusion parameters of silicone mixes studied, determined at different screw rotational speed**
TABELA 2. **Parametry procesu wytłaczania badanych mieszanek silikonowych przy różnych prędkościach obrotowych ślimaka**

A) mix A

10 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	4.8	2.7	4.6	19.0	4.2	19.7
2.	5.0	2.9	4.8	20.0	4.0	21.7
3.	4.6	2.6	4.4	18.0	4.3	20.7
4.	4.5	2.5	4.2	18.0	6.7	18.7
5.	4.7	2.7	4.5	19.0	4.3	22.7
Average value				**19.0**	**4.7**	**20.7**
30 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	13.1	7.7	13.0	52.0	0.8	25.1
2.	13.0	7.7	12.9	52.0	0.8	25.2
3.	12.7	7.5	12.5	51.0	1.6	25.6
4.	13.5	7.9	13.3	54.0	1.5	24.3
5.	13.0	7.8	12.9	52.0	0.8	27.7
Average value				**52.0**	**1.1**	**25.6**
50 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	20.7	12.9	20.5	83.0	1.0	32.4
2.	20.8	13.0	20.5	83.0	1.4	33.3
3.	21.2	13.4	21.0	85.0	0.9	34.5
4.	22.0	13.5	21.9	88.0	0.5	30.2
5.	21.8	13.5	21.6	87.0	0.9	31.7
Average value				**85.0**	**0.9**	**32.4**

B) mix B

10 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	4.5	2.9	4.0	18.0	11.1	136.1
2.	6.0	3.9	5.5	24.0	8.3	137.3
3.	5.0	3.0	4.5	20.0	10.0	126.7
4.	5.0	3.2	4.5	20.0	10.0	135.2
5.	4.5	2.9	4.0	18.0	11.1	136.1
Average value				**20.0**	**10.1**	**135.1**
30 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	9.5	6.5	9.0	38.0	5.3	144.6
2.	9.5	6.6	9.0	38.0	5.3	146.8
3.	9.0	6.2	8.2	36.0	8.9	145.6
4.	9.5	6.5	9.0	38.0	5.3	144.6
5.	8.0	5.5	7.5	32.0	6.3	145.3
Average value				**36.0**	**6.2**	**145.4**
50 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	11.5	8.2	10.5	46.0	8.7	150.7
2.	12.0	8.5	11.0	48.0	8.3	149.7
3.	13.0	9.2	11.5	52.0	11.5	149.6
4.	12.0	8.5	11.0	48.0	8.3	149.7
5.	12.0	8.5	11.3	48.0	5.8	149.7
Average value				**48.0**	**8.6**	**149.9**

C) mix C

10 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	1.9	1.1	1.5	8.0	21.1	22.1
2.	1.7	1.0	1.2	7.0	29.4	22.7
3.	1.9	1.1	1.3	8.0	31.6	26.5
4.	1.8	1.0	1.3	7.0	27.8	22.9
5.	1.8	1,1	1.4	7.0	22.2	26.5
Average value				**7.0**	**26.4**	**24.1**
30 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	4.0	2.5	3.5	16.0	12.5	33.0
2.	4.2	2.6	4.0	17.0	4.8	31.2
3.	4.5	2.8	4.4	18.0	2.2	31.9
4.	4.3	2.7	3.5	17.0	18.6	31.6
5.	4.0	2.5	3.6	16.0	10.0	33.5
Average value				**17.0**	**9.6**	**32.2**
50 rpm						
No.	l_{15} [cm]	g [g]	l_{30} [cm]	V_w [cm/min]	S_L [%]	W_p [%]
1.	10.0	6.5	9.3	40.0	7.0	38.5
2.	8.7	5.7	7.6	35.0	12.6	38.4
3.	7.9	5.1	6.9	32.0	12.7	37.4
4.	8.0	5.2	7.0	32.0	12.5	37.0
5.	9.0	5.8	8.1	36.0	10.0	37.8
Average value				**35.0**	**11.0**	**37.8**

The tremendous difference, this time concerning material B, is associated with extrusion swelling. The Barrus' effect for mix B – W_p, increases significantly with an increasing rotational speed, stabilizing eventually at an unacceptable level of c.a. 150%.

Pictures of some extrudates of mix A, illustrating the edge defects appearing at a low screw rotational speed, characteristic for all the mixes studied, are given in Figure 3.

The optimal extrusion conditions for the mixes studied are very close to each other. The higher screw rotational speed the better. The only difference concerns the width of the so-called "extrusion window". A lower screw rotational speed, typically not exceeding 60 rpm for A and 90 rpm for B materials, results in the appearance of edge defects, whereas a French mix can be extruded with a lower rotational speed without any problem concerning the quality of the extrudate. Visual inspection of the extrudates according to ASTM D 2230-83 - A and B standards, suggests that extrusion is the most effective (A = 4 and B = 9÷10) at the temperature of head 50°C and channel die of 60°C, for all the materials studied. The vulcametric parameters and mechanical properties of the ceramizable silicone composites studied are given in Table 3.

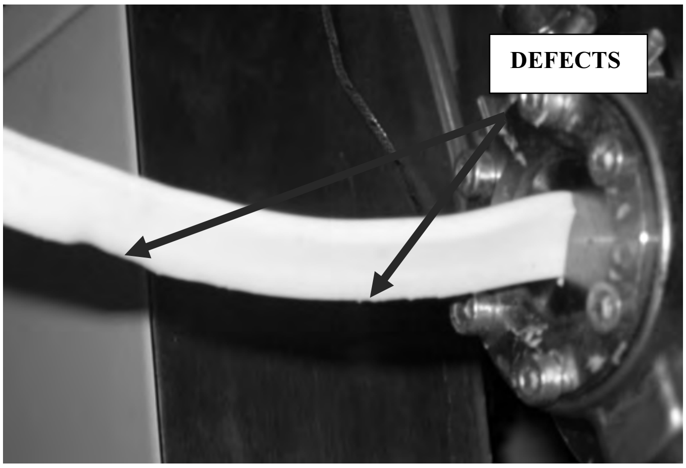
Similar to the extrudability, one year storage of the mixes studied practically did not influence the vulcametric parameters or mechanical properties of the composites prepared by their crosslinking with 2,4 (di)chloro benzoyl peroxide. On the example of composite A, its value of TS decreases only by c.a. 1%, TES by c.a. 5% and E_B by 12%.

TABLE 3. **Vulcametric parameters and mechanical properties: tensile strength (TS) and tear strength (TES) of ceramizable silicone composites studied**

TABELA 3. **Parametry procesu wulkanizacji i właściwości mechaniczne: wytrzymałość na rozciąganie (TS) i wytrzymałość na rozdzieranie (TES) badanych kompozytów ceramizujących**

Composite	A	B	C
Vulcametric parameters			
M_{min} [dNm]	11.0	15.8	13.5
M_{max} [dNm]	108.4	66.1	90.6
ΔM [dNm]	97.4	50.3	77.1
$M_{opt.}$ [dNm]	98.7	61.1	99.2
$\tau_{0.2}$ [s]	24	41	20
$\tau_{0.9}$ [s]	70	99	71
Mechanical properties			
S_{100} [MPa]	4.5	2.2	5.6
S_{200} [MPa]	7.1	4.4	-
S_{300} [MPa]	-	6.3	-
TS [MPa]	8.3	7.3	7.7
E_B [%]	254	393	157
Hardness [°Sh A]	71	62	71
TES [kN/m]	18.0	15.2	16.0

A) extrusion with screw rotational speed of 60 rpm



B) extrusion with screw rotational speed of 200 rpm (lack of defects)

Fig. 3. Pictures showing the influence of screw rotational speed on quality of mix A extrudates

Rys. 3. Przedstawienie wpływu prędkości obrotowej ślimaka na jakość wytłoczyn z mieszanki A

SUMMARY AND CONCLUSIONS

Despite similar rheological characteristics, $\eta = f(\gamma_R)$, in the range of shearing rate 10÷140 $s^{-1}$of the silicone composites studied, mix A processes the best among other commercial mixes. It manifests itself in a significantly higher extrusion rate - more than doubled in comparison to mix C, accompanied by the lowest Barrus' effect - almost four times lower in comparison to mix B, for the highest extrusion rate tested (50 rpm). Additionally, material A is practically free from linear shrinkage, which in the case of material C reaches above 10%. The higher the screw rotational speed the best efficiency of extrusion. The quality of the extrudates is also better at a higher extrusion rate. There are also small differences in the range of the extrusion window between the mixes studied. A and especially B mixes have to be extruded with a slightly higher speed (above 60 and 90 rpm respectively) in comparison to composite C. The apparent discrepancy between the rheological data and extrusion characteristics confirms that the former is the only necessary condition required for good processing of polymer composites, whereas the final condition is the good extrudability characteristics of the material. The mechanical properties of the crosslinked composites follow their vulcametric characteristics. The best one regarding the TS and TES values is composite A, however, all of them meet in excess the requirements of the cable industry.

The composition of the mineral phase, among the glaze and silicone rubber matrix, is responsible for the differences in the extrudability of the composites studied. Composites containing small particles of milled quartz and wollastonite dominate over the one based on large particles of mica and zinc oxide regarding the processing parameters.

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