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The Effect of Temperature on the Parameters of the Mixing Process in the Preparation of Elastomeric Composites

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Abstract

Elastomeric composites are among the key engineering materials that find wide application in industrial and domestic applications. The work deals with the preparation of elastomeric composites based on natural rubber (NR) filled with 20 phr cellulose (CEL). The reason for preparing a composite filled with biopolymer is the environmental point of view, to replace synthetic polymers at least partially with natural materials. When using CEL in an elastomeric matrix, high processing temperature can cause problems, leading to degradation of the CEL. The solution could be the preparation of polymer composites at lower temperatures. The influence of processing temperature (50 – 90 °C), torque (MT) during the preparation of elastomeric composites filled with CEL was investigated. Rheological tests made it possible to determine the optimal processing parameters and properties of elastomeric composites filled with CEL. Hardness, mechanical properties were also studied on the prepared NR/CEL composites.

Keywords

Temperature, Composites, Elastomer, Natural rubber, Torque

Introduction

The resulting properties of prepared blends and composites depend not only on the properties and quantity of their basic components, but also on the method and conditions of their preparation. A critical process variable in the entire mixing process is temperature, which affects the quality and efficiency of the preparation of elastomeric blends and composites. Effective temperature control plays a key role in achieving the desired properties of composites [1]. Among the main effects of temperature on the entire elastomeric blend's preparation process is the influence of the environment temperature and the temperature of the functional equipment components in which the blends are prepared. Heating and insulation of functional components includes heating the main parts of the blend preparation equipment, the mixing part to ensure the working viscosity suitable for the correct composite or blend, thereby regulating the environment of the whole process in each range [2]. Mixing operations have a direct effect on mixing efficiency and macro dispersion. In general, a better dispersion of the filler is achieved by longer and more intense mixing. Longer and more intense mixing, however, leads to degradation of the bio filler and the elastomeric matrix in which the filler is dispersed. This is especially problematic for NR, which is sensitive to mechanical/thermal degradation [3].

In this work, the influence of temperature and the method of mixing components during the preparation of elastomeric composites filled with CEL GW 70 (20 phr) were investigated. In previous published works [4-6], less attention was paid to the study of the mixing process and the influence of temperature,

which determine the correct mixing method. The influence of temperature and MT during the preparation of composites (NR and CEL GW 70) was evaluated through the mechanical properties and morphology of samples of prepared elastomeric composites based on NR filled with GW 70 CEL.

Materials and methods

Elastomeric blends for this study were prepared in 80 cm^3 chamber in universal laboratory mixer Brabender Plastograph EC plus (Brabender, Duisburg, Germany). All blends were prepared according to one recipe table 1 to avoid the undesirable influence of the blend's composition on the measurable parameters of the mixing process and the tested properties of the blends. To make the influence of the mixing temperature on the properties of the blends visible, a one-stage mixing process was chosen. The set mixing temperature was different for each blend in the interval from 50 to 90 °C with a step of 10 °C. During the mixing process, the real temperature of the mixed blend (RTB) as well as the MT were recorded using the Winmix software. Additional homogenization of the blends after mixing was performed on a laboratory two-roll mill LaboWalz W150 (Voght Labormaschinen GmBH, Berlin, Germany). Before performing further tests, the blends were left to stand for 24 h at laboratory temperature. Vulcanization characteristics and rheological properties were determined at a temperature of 160 °C on a RPA 2000 device (Alfa Technologies Ltd., Akron, OH, USA). With an oscillation angle of 1° and a frequency of 1.67 Hz. Type A2 (ISO 37) specimens with a total length of 75 mm were prepared using the punching method on an Instron cutter machine (Instron, Norwood, MA, USA). A Shimadzu AG-X plus universal tensile tester (Shimadzu, Tokyo, Japan) was used to determine the tensile properties of the prepared vulcanizates. The test speed was 500 mm/min, and the gauge length (distance between the jaws) was 50 mm.

Results and discussion

The first studied property in the work is the MT recorded during the mixing process. From the course of the MT curve, it is possible to see the change in the viscosities of the raw materials during the mixing process as well as the change in the shear forces corresponding to the addition of individual raw materials [7]. The beginning of the mixing process, specifically the entry of the matrix with the need for plasticization into the mixing process, is manifested by a sharp increase in the MT value. The matrix enters the mixing process with a high viscosity unaffected by temperature (without preheating, stored at laboratory temperature). After the end of the matrix dosing phase, the curve reaches a maximum, which corresponds to the beginning of the plasticization process. From figure 1 with increasing mixing temperature, the values of the maximum peak of the MT curves corresponding to the end of the dosing phase decrease. A higher mixing temperature causes faster overheating of the volume of the dosed matrix in the chamber of the mixing device, and therefore also an acceleration of the decrease in the viscosity of the matrix. The matrix plasticization itself begins precisely when the maximum of the MT curve is reached.

Table 1: Materials used for preparation of elastomeric blends.

Material	Content (phr)	Producer
SMR 10	100	Lee Rubber, Kuala Lumpur, Malaysia
GW 70	20	Greencel Ltd. Company, Hencovce, Slovakia
Zinc oxide	2	SlovZink, a.s., Košeca, Slovakia
Stearic acid (SA)	1.5	Setuza A.s., Ústí nad Labem, Czech Republic
N-tert-butyl-2-benzothiazole sulfenamide (TBBS)	1.0	Duslo, a.s., Šaľa, Slovakia
Sulphur	1.5	Eastman Chemical company, Kingsport, TN, USA

In plasticization, the purpose is to achieve softening of the matrix (decrease in viscosity) caused by breaking of its chains [8, 9]. That is the reason, why plasticization on the MT curve is manifested by a gradual decrease in the MT value. Again, the influence of the mixing temperature is visible, where, similarly to the case of the peak maximum during matrix dosing, the decrease in the MT value is faster at higher temperatures. Higher temperature causes more intensive breaks of rubber chain due to the rotation of the mixing device blades and the plasticization of the matrix itself therefore takes place in a shorter time. In this study, the influence of the mixing temperature on the properties of the prepared mixtures was investigated and for this reason, all raw materials had a given (equal) mixing time without considering the mixing temperature, to avoid changes due to the adjustment of the mixing time of individual raw materials. The process of plasticization of rubber matrix can be considered completed when the MT values oscillate around an imaginary horizontal line. The effect of another raw material, zinc oxide, is not significant, since its content in the blends is not sufficient to change the trend of the MT curve. The raw material with a significant impact on the MT curve is the filler-CEL. Its content in the blend is the second highest (after the matrix) and its presence in the blend after dosing increases the viscosity. The CEL content is completely dosed into the dosing slot of the mixing device, and the filler is drawn through the slot directly into the blend. The blend gradually draws parts of the CEL volume through the slot, causing a sudden increase in the viscosity and stiffness of the blend, which causes a momentary stop in the dosing of filler into the blend and a decrease in the MT curve. After mixing part of the filler in the volume of the matrix,

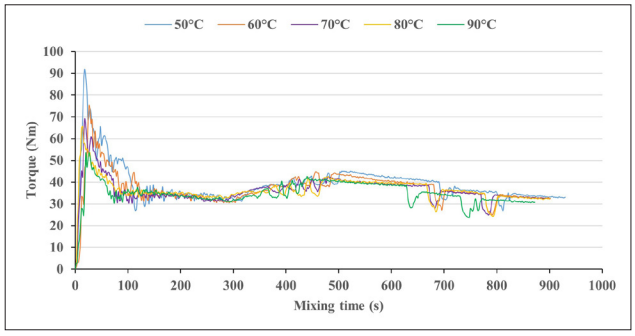


Figure 1: MT.

the viscosity will drop again and the stiffness of the blend will decrease, especially in the area under the slot of the mixing device, which will cause part of the filler volume to be drawn in again and the MT values to increase. These two repetitive operations create characteristic V-shaped peaks on the MT curve. As can be seen from the graph in figure 1, the number of peaks depends mainly on the viscosity of the matrix and the ability to overcome the impact hardening when receiving part of the filler content. With increasing mixing temperature, the number of peaks decreases, and the time required to dose the entire filler volume into the blend is shortened. By shortening the filler dosing interval, it is also possible to shorten the total mixing time of the blend, however, in the case of the blends in this study, the same raw materials have the same mixing time in all prepared blends, the shorter time is precisely the effect of the filler dosing. Another raw material added to the mixing process is SA, which is referred to in many publications as a dispersing agent or softener and during the mixing process, depending on the content in the blend, it shows a specific sign on the MT curve-the shape of the letter V. SA is a raw material with a low melting point and, after dosing into the elastomer blend, it will suddenly reduce the viscosity of the blend as well as the friction between the polymer chains. After dispersing SA in the blend volume, the MT value increases, but does not reach the original value, since it remains in the blend volume between the matrix chains [10, 11]. After the addition of SA and the formation of a V-shaped peak, it is seen how the mixing process is shortened due to the increase in the mixing temperature (the peak shifts towards lower mixing times), especially by faster filler dosing. The last raw materials dosed in the mixing process are the vulcanizing agent and the vulcanization accelerator, i.e., the curing system, to prevent possible scorching of the blend due to the real temperature [12]. Since the blends already contain the raw materials constituting the majority of the volume, opening the mixing chamber causes a release of pressure and a momentary decrease in the MT value. Again, the change due to the increase in the mixing temperature and the shift of the curing system dosing time to lower values is clearly visible.

The second parameter recorded during the mixing process is the RTB in the mixing chamber, which directly affects the processes in the blend (breaks of rubber chain, viscosity, etc.) [13]. The course of the RTB is different from the MT curve, since its value depends not only on the type of dosed raw materials, but also on the processes taking place in the blend. The friction of the rubber chains in the blend due to the rotation of the mixing device blades occurs during the entire mixing process and has an impact on the RTB curve. From the analysis of the curves in figure 2, at the beginning of the mixing process, i.e., the dosing of rubber into the mixing device chamber, a decrease in the RTB value occurs. This decrease is caused by the same factor that, on the contrary, increases the MT value, namely the input temperature of the rubber and the sudden cooling of the temperature sensor due to it. With increasing temperature, the decrease in RTB is more pronounced and the time required to reach the original (set) mixing temperature is extended. Rubber at laboratory temperature absorbs heat from the environment, in this case from the surfaces of the

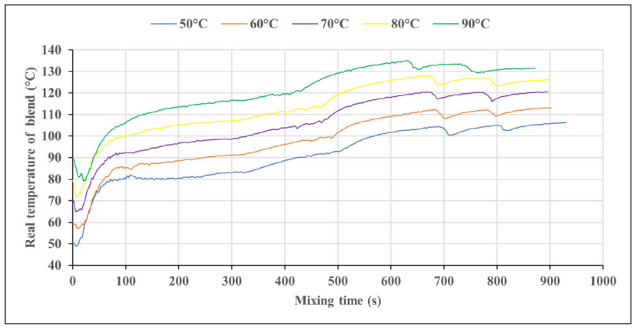


Figure 2: RTB.

mixing chamber and the blades. The higher the temperature difference between the rubber and the mixing chamber, the more pronounced the initial cooling effect will be. The increase in the time required to reach the set temperature again, after the initial cooling, is related to energy compensation [14, 15]. The higher the set mixing temperature, the longer the system needs to reach equilibrium, in this case the set mixing temperature. As a result of the blade speed and the mixing chamber temperature, the viscosity of the rubber starts to decrease and the frictional forces between the rubber chains and the shear forces caused by the blades speed come to the fore, causing the RTB curve to increase. Depending on the set mixing temperature, the development of the curve in the mixing interval of 100 - 300 s changes. With increasing mixing temperature, the curve in the mentioned interval becomes steeper, which is caused by increased shear force due to the rotation of the mixing device blades (higher efficiency) and at the same time higher internal friction force (this effect is not visible on the MT curve). Another steep increase is visible after adding filler, which increases the viscosity of the mixed blend as well as the shear force in the mixing chamber. Adding filler again changes the steepness of the RTB curve, but this time the effect of the set mixing temperature is opposite to that in the case of rubber. The steepness of the curve in the interval of filler dosing into the blend (300 - 400 s) is highest at the lowest mixing temperature and decreases with its increase. At low mixing temperatures, the break of rubber chains is not so intense and therefore longer chains remain in the rubber volume, which, when adding filler, will significantly increase the shear forces in the mixing chamber compared to shorter rubber chains at higher mixing temperatures [16]. With increasing mixing temperature, the time interval corresponding to the dosage of the filler is also shortened (visible also on the MT curves). The raw material SA has a significant influence on the RTB curve, the effect of which is clearly visible also on the MT curve, and after its addition a decrease occurs. This decrease in the RTB value is caused by a decrease in the shear force as well as the internal friction of the rubber chain precisely with the help of SA. However, by intercorporate SA into the volume of the blend, the RTB increases slightly. The last significant change and therefore the decrease in RTB is caused by the influence of the opening of the chamber, the release of pressure and thus the shear forces in the rubber mixing chamber. This decrease occurred before the actual addition of the curing system to the blend [17].

Figure 2 shows if the set mixing temperature is higher, the higher the RTB curve values reaches. However, if we want to examine the real increase in the temperature value depending on the set one, it is necessary to calculate the difference between the set temperature and each RTB point (ΔT). Such a difference is graphically shown in figure 3 and the lower the set mixing temperature, the higher the difference ΔT. We can justify this effect by the just set temperature and its effect on the rubber in the first phase of mixing (slower heating of the rubber volume and the associated higher resistance to the rotation of the blades visible on the MT curve) as well as after adding filler to the blend (longer rubber chains and a more significant increase in shear force) [18].

Rheological properties

After the mixing process, rheological properties table 2 were studied on the prepared blends, namely the minimum (M_L) and maximum torque (M_H) (Figure 4). The M_L corresponds to the stiffness of the unvulcanised blend and from its value it is possible to determine the influence of previous processes, especially mixing. The lowest value was achieved by the blend with a mixing temperature of 50 °C, mainly due to the low intensity of rubber chain breaks and therefore the content of longer rubber chains, which have greater flexibility compared to short ones. By increasing the mixing temperature up to 80 °C, an increase in M_L values is seen, which corresponds to the shortening of the rubber chain length (discussed in the work above) and therefore the reduction of flexibility and thus the increase in stiffness of the unvulcanised blend. The change in trend occurs at a mixing temperature of 90 °C, where the M_L value decreased. This decrease may be caused by excessive breaking of the rubber chain (possibly also referred to as degradation) as well as by the breakage of filler fibers due to high shear force and internal friction force in mixing chamber [19]. The M_H values also have a similar trend to the M_L, with the increase in the value in the interval 50 to 70 °C being more pronounced. Shortening the rubber chains creates additional spaces for higher crosslinking of the samples, which increases the MT. At the same time, the filler fibers are more broken during the mixing process, thus reducing the barrier for the formation of crosslinks. After the mixing temperature exceeds 70 °C, the M_H value for the blends begins to decrease. If the blend undergoes an intensive rubber break chain during mixing, the rubber chains are significantly shortened, which can have a negative effect on the density of the resulting vulcanization network during the vulcanization process. With a short rubber chain, a situation may arise where the vulcanization system and its content are not sufficient to create a denser network during the vulcanization process, which leads to a decrease in the M_H value. In addition to the rubber chain, the density of the vulcanization network is also affected by the filler itself and the length of its fibers, which is shortened more intensively with increasing temperature [20].

Curing characteristics

Curing characteristics in table 3 are properties that directly affect the length of the final product production process as well as the economic costs associated with this process (Figure 5).

The first of the curing characteristics is scorch time (t_{s02}),

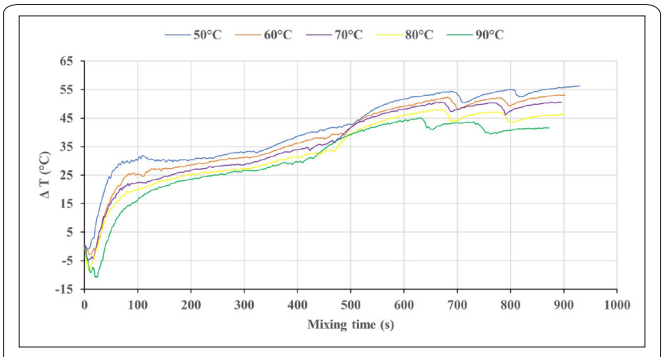


Figure 3: Difference between set temperature and RTB.

Table 2: Rheological properties of blends.

Mixing temperature (°C)	M_L (dNm)	Standard deviation (dNm)	M_H (dNm)	Standard deviation (dNm)
50	1.5	0.030	15.4	0.154
60	1.85	0.034	16.05	0.161
70	1.92	0.038	16.44	0.160
80	2	0.040	16.4	0.162
90	1.84	0.037	16.31	0.163

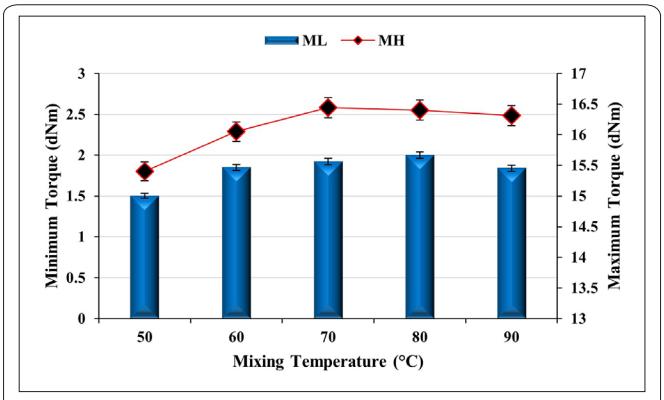


Figure 4: Rheological properties.

Table 3: Curing characteristics of prepared blend.

Mixing temperature (°C)	t_{s02}	Standard deviation (dNm)	t_{c90}	Standard deviation (dNm)
50	2.93	0.058	5.43	0.109
60	3.16	0.063	5.15	0.103
70	3.46	0.069	5.46	0.109
80	3.6	0.070	5.81	0.116
90	3.27	0.066	5.34	0.107

which is the beginning of the vulcanization process. Its value is important from the point of view of pressing the shape of complex products due to the necessity of filling the Mold in the entire volume even before the vulcanization network begins to form [21, 22]. In the interval from 50 to 80 °C, a continuous increase in the t_{s02} value can be seen (similar trend as in the case of M_L) caused by the shortening of the rubber chain as well as the length of the filler fibers and therefore the need to create a higher number of cross-links to change the value of the MT, which is the basis for the calculation of scorch time, which increases the time required for this operation. In

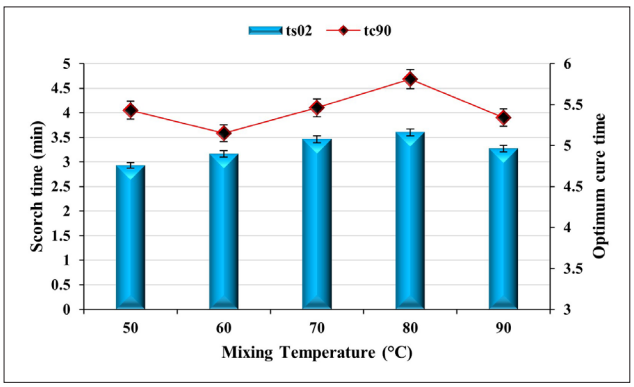


Figure 5: Curing characteristics.

the case of a mixing temperature of 90 °C, it can be assumed that the increased RTB and the holding at this temperature during the mixing process activated the vulcanization accelerator TBBS, which is known for its delay effect, and part of the delay cycle began to run already during the mixing process. As a result, after reheating the blend to the vulcanization temperature, the accelerator delay time was no longer the same as for blends with a lower mixing temperature and, despite the short rubber chain, the blend achieved a decrease in the t_{s02} value [23].

The optimum cure time (t_{c90}), i.e., the time required for the formation of 90% of the vulcanization network, was reduced by increasing the temperature by 10 °C (in the case of the blend with a mixing temperature of 60 °C) compared to the blend with the minimum mixing temperature. This decrease can be justified by an increase in the vulcanization rate (from the point of view of t_{c90}, the optimal ratio of rubber chain length and curing accelerator activity) [24]. Further increase in the mixing temperature above 60 °C leads to an increase in the t_{c90} value up to a mixing temperature of 80 °C. Since the blends in this interval have shortened rubber chains and filler Fibers due to the intensity and temperature of blend by mixing process, the vulcanization rate decreases and during the vulcanization process it is necessary to create a larger number of sulphur bridges between short rubber chains (with a decreasing barrier formed by the length of the filler fibers), which extends the required time. For the blend with a mixing temperature of 90 °C, the t_{c90} value decreased, like the case of t_{s02}. The decrease is mainly caused by the reduction of scorch time and thus the rapid onset of the vulcanization process of the blend. If we compare the difference between t_{c90} and t_{s02} of blends with mixing temperature of 70 °C and 90 °C, we find that they are almost identical (2 min, 2.07 min), however, the decrease in t_{s02} caused a decrease in t_{c90} in the case of the blend with mixing temperature of 90 °C [25].

Hardness

The hardness values table 4 of the prepared blends follow the trend of the M_H values (Figure 6). Although the M_H values of the blends mixed at 50 °C and 60 °C were different, the ΔM value and therefore the difference between the M_H and M_L values (often referred to as the vulcanization efficiency) are important, which results in a minimal change in the ΔM value for these blends (13.9 and 14.2 dNm), which is reflected in

Table 4: Hardness of prepared blends.

Mixing temperature (°C)	Hardness (ShA)	Standard deviation (ShA)
50	58.45	0.68
60	58.50	0.32
70	59.58	0.73
80	57.93	0.29
90	56.43	0.56

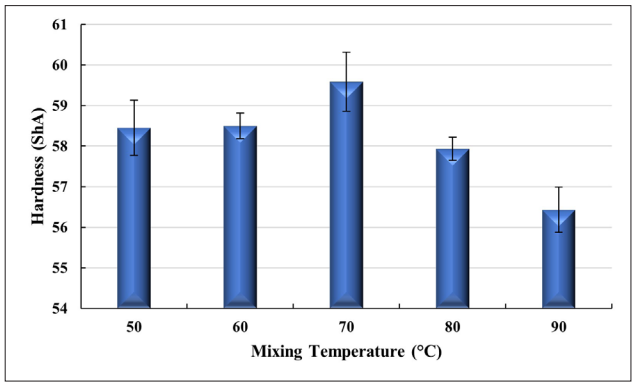


Figure 6: Hardness of prepared blends.

the hardness value. The highest hardness was achieved by the blend mixed at 70 °C, which was also predicted by the M_H value. From the point of view of hardness, this blend is a perfect synergy of rubber chain break and filler stiffness effect. Shorter rubber chains provide a higher possibility of vulcanization network density as well as the manifestation of the stiffening effect of the filler, whose fibers are shortened during the mixing process.

A further increase in the mixing temperature (80 °C and 90 °C) already causes a decrease in the hardness of the blend. The downward trend is also seen in the M_H values, and it can be assumed that it is caused, as with M_H, by the crosslinking density and the length of the rubber chains, where due to the short rubber chain, even the filler does not achieve a significant reinforcing effect [26, 27].

Tensile properties

The measured results of the tensile properties table 5 study showed that the least affected by the mixing temperature are the elongation values. The change in values is at a maximum level of approximately 4%, and it is not possible to clearly describe the trend of the values (Figure 7).

Elongation at break (EaB) is one of the properties of elastomeric vulcanizates dependent on the density of the vulcanization network formed, the length of the rubber chains and the possibility of their motion, as well as the reinforcing effect of the filler used. It can be seen that the blend with the highest crosslinking (70 °C) did not reach the highest value, on the contrary, the blend with a lower M_H value and therefore also crosslinking as well as less intensive shortening of the filler fibers and rubber chain during the mixing process (60 °C) as well as the blend with more intensive rubber chain and filler fibers break during the mixing process and a decrease in M_H

Table 5: Tensile properties of prepared blends.

Mixing temperature (˚C)	Tensile strength (TS; Mpa)	Standard deviation (Mpa)	EaB (%)	Standard deviation (%)
50	11.8	0.37	650.45	18.41
60	13.18	0.2	670.74	12.08
70	13.07	0.3	649.47	18.76
80	13.42	0.34	668.64	9.63
90	12.99	0.07	636.12	2.81

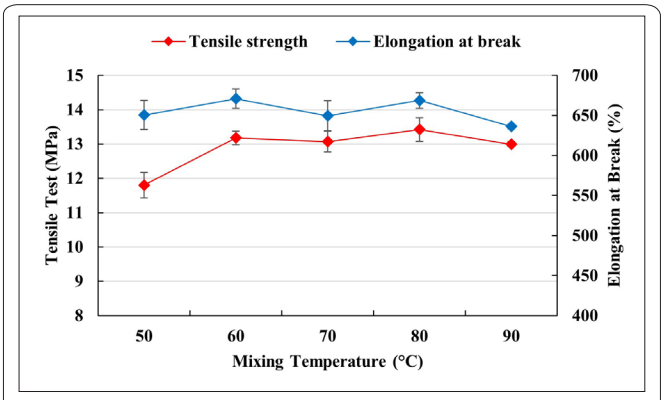


Figure 7: Tensile properties of prepared blends.

(80 ˚C) achieved a higher EaB value. The high density of the crosslink network, as well as the length of the rubber chain and filler fibers affect the possibility of motion of the rubber chain, which negatively affects the EaB. For long rubber chains, a lower crosslinking of the blend is more advantageous, which preserves flexibility (60 ˚C). On the contrary, for short chains, a decrease in the crosslinking density is more advantageous, since the sulphur bridges themselves also affect the motion of the rubber chain (80 ˚C). The maximum mixing temperature reached the minimum EaB value, which is caused by the high crosslinking of the sample with respect to the length of the rubber chain (limitation of the motion of the rubber chain under tensile stress also due to the length of the filler fibers) [28, 29]. The tensile strength (TS) values of the individual blends show similar behaviour as in the case of EaB. The difference is the increase in the TS value by increasing the mixing temperature from 50 ˚C to 60 ˚C, which is almost 12%. By further increasing the mixing temperature (60 ˚C – 90 ˚C), the changes in the TS value of the blend were no longer so significant (about 3%). The highest value was achieved by the blend with a mixing temperature of 80 ˚C, which can be attributed from the point of view of TS to the optimal density of the vulcanization network to the length of the rubber chain. An interesting finding is that the blend with a mixing temperature of 70 ˚C, which recorded a decrease in the EaB value, shows a decrease in the TS value again compared to the blends of 60 ˚C and 80 ˚C. The highest density of the vulcanization network in combination with the length of the rubber chain caused a restriction of the rubber chain motion (reduction of EaB) and brittleness of the blend (reduction of TS) [30].

Conclusion

The results of this study confirm that the mixing temperature has a significant impact on the rheological and mechanical properties of elastomeric blends based on CEL -filled NR (SMR10 and 20 phr GW 70). With increasing mixing temperature, the M_L decreases from 1.16 dNm at 50 ˚C to 0.87 dNm at 90 ˚C, which indicates more efficient plasticization of the blend and a decrease in viscosity. At the same time, the optimum vulcanization time (t_{c90}) is shortened from 5.7 min to 4.2 min, which means faster processability of the material in the production process. Mechanical properties, especially TS, reached the highest value of 16.2 MPa at the optimal mixing temperature (70 ˚C), while this value was lower at both lower and higher temperatures. The hardness of the blends ranged from 52 – 56 ShA and was only minimally affected by temperature. The EaB reached a maximum of 410% at 70 ˚C, which proves that the correct choice of mixing temperature allows optimizing the elasticity of the material. Mixing temperature affects the length of the rubber chain, filler fibers and consequently the density of the vulcanization network formed during the vulcanization process. For some properties, a blend containing long rubber chains is more advantageous and therefore a mixing process with a lower temperature, for others, more significant mixing in terms of breaks of rubber chain, on which the density of the vulcanization network itself also depends. The main contribution of the work is the experimental confirmation that by choosing the appropriate mixing temperature, it is possible to selectively influence the structure and properties of bio composites based on NR (SMR 10) and CEL (20 phr GW 70) without the need to use traditional synthetic fillers. The work also shows that reducing the mixing temperature contributes to protecting the bio-filler from thermal degradation, thereby maintaining its functionality and improving the environmental profile of the resulting material. Optimizing the mixing temperature leads to higher TS and EaB, while at the same time shortening the vulcanization time and increasing production efficiency. This knowledge is important not only for the development of more environmentally friendly rubber materials, but also for industrial practice, where it is necessary to combine high utility value with environmental sustainability. The results of this work thus open new possibilities for the use of renewable raw materials in the rubber industry and provide specific recommendations for the effective preparation and processing of elastomer blends with bio-fillers (e.g., study of influence of mixing speed, order of adding materials in mixing process, dosage of CEL in mixing process etc.).

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Conflict of Interest

None.

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