The use of illite in function of filler applied in rubber blend

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Abstract. The presented work was dealing with the study of the commercial filler influence change in rubber blend by an alternative filler based on the clay mineral - illite. The focus of the presented work was aimed at the study of selected curing characteristics of rubber blend with addition of clay mineral filler and physico-mechanical properties of prepared vulcanizates. Curing characteristics, the processing safety, minimum and maximum torque, optimal curing time and curing rate coefficient were determined during the curing experiment phase. Selected physico-mechanical properties were given by the determination of hardness, tensibility and tensile strength. The obtained results proved the possibility of partial commercial filler replacement by an alternative filler and the positive effect of clay mineral on resulting important properties in rubber industry.

1. Introduction

Commercial fillers are solid state substances with particles of very small fractions affecting resulting properties of a rubber blend under an appropriate dispersibility condition. Commercial carbon black fillers increase stiffness, tensile strength, aging, corrosion and heat resistance, toughness and abrasion resistance. After all, these fillers affect the final appearance and cost of the product. Efficiency of fillers depends on their structure and particles size and shape, surface activity and on the capability of fillers to chain with the rubber matrix. Fillers are usually added to the blend in the form of short fibres or in a powder form, to ensure the decrease of dustiness are granulated. The basic type of rubber fillers is carbon black improving mainly abrasion resistance and tensile strength. The important knowledge of carbon black is the humidity content [1–10].

Bright fillers are non-carbon types of fillers used in rubber industry. This group includes inorganic fillers applied to reduce the cost and achieve the white color of the product, SiO₂, kaolinite, illite, montmorillonite and other clay minerals etc.

Clays are hydrated aluminosilicates with layered structure or phyllosilicates with expanding structure and can be clarified as nanomaterials [11–13]. Illite is firmed by mica and smectite weathering of volcanic glass. It is a plastic clay non-expanding mineral in white, grey or yellow color. Illite belongs to the group of mica with an interlayer defect of structural type 2:1. Its composition is between smectite and muscovite. Structure of illite contains Mg²⁺, Si⁴⁺, K⁺, Al³⁺ cations [11–17].

Under the term modification of clay mineral illite is meant the intercalation of the clay mineral structure when ions, atoms, complex ions, molecules and other substances are inserted into the interlayer space between the two layers of the clay mineral. The aim of modification is to change physical properties of the clay mineral such as: to change the interaction of the clay mineral between individual layers, improve the interaction between the clay and polymer matrix, to increase the interlayer space and to modify the properties of the clay mineral [18, 19].
The subject of the submitted work is the study of the properties of modified clay products, which were applied to rubber blends as fillers. The study was performed by IR-spectroscopy and X-ray diffraction analysis. The prepared clay products were applied to the tread rubber blends as fillers. Their influence on the curing characteristics of the blends was studied. Their impact on the physical-mechanical properties before and after aging and the rebound for the preparing vulcanizates were evaluated.

2. Experimental

2.1. Preparation of modified monoionic forms of illite product
Monoionic forms of illite Co$^{2+}$, Cu$^{2+}$, Ni$^{2+}$ (Figure 1) were prepared from commercial illite product dried in an oven at 60 °C by a saturating the solutions of metal acetates with a concentration of 0.5 mol.dm$^{-3}$. pH was adjusted by a solution of NaOH with a concentration of 0.3 mol.dm$^{-3}$. Prepared samples of monoionic forms were then dried in an oven, shred and sieved through 0.025 mm sieve eye.

![Figure 1. Preparation of monoionic modified forms.](image)

2.2. Characterization of monoionic forms
To characterize the commercial clay products and its modified forms were used x-ray fluorescence energy-dispersive spectroscopy, thermal analysis, infrared spectroscopy and x-ray diffraction powder analysis.

X-ray fluorescence energy-dispersive spectroscopy was performed by a spectrometer Shimadzu EDX-7000 at Faculty of Industrial Technologies in Púchov. Thermal analysis was performed in the range of 20 °C to 1000 °C at the instrument TGA/DSC 2 STAR$^\circ$ system METLER TOLEDO.
analysis was carried out in nitrogen atmosphere. Infrared spectroscopy was done by an infrared spectrometer Nicolet iS50 FT-IR in the middle infrared area 4000 cm\(^{-1}\) – 400 cm\(^{-1}\) by ATR technique. X-ray diffraction powder analysis was performed by a PANALYTICAL EMPYREAN device.

2.3. Characterization of prepared rubber blends
Characterization of rubber properties was performed on model tread blend. Rheological and curing characteristics were determined (minimal and maximal torque, safety processing, optimal curing time and curing rate coefficient), physical and mechanical properties (tensile strength, elongation at break, hardness), rebound and properties after accelerated thermal aging according to standards.

2.4. Used devices
Rubber tread blends were prepared in a kneading machine Brabender Plastograph EC Plus at the temperature of 120 °C in the first mixing stage and in the temperature of 100 °C in the second mixing stage at 50 rpm. Homogenization was performed on the double cylinder Labo Waaltz. Curing characteristics were measured at the Rheometer RPA 2000 device by Alpha Technologies at the temperature of 160 °C for 30 min. Rubber blends were cured in the curing press Fontjine LabEcon 600 2 mm thick to determine rebound. Physical and mechanical properties were determined on Shimadzu AG-X Plus device.

3. Results
Monoionic forms of clay mineral illite (ILCo, ILCu, ILNi) were characterized by x-ray florescence energy-dispersive spectroscopy, infrared spectroscopy, x-ray diffraction powder analysis and thermal analysis. The content of cobalt, nickel, cooper from monoionic Co\(^{2+}\), Cu\(^{2+}\), Ni\(^{2+}\) forms - ILCo, ILCu and ILNi (Table 1) and content oxides of initial illite product IL, were determined by x-ray florescence energy-dispersive spectroscopy (Table 2).

| Sample  | Co (%)     | Cu (%)     | Ni (%)     |
|---------|------------|------------|------------|
| ILCo    | 13.31 ± 0.02 |            |            |
| ILCu    |            | 18.43 ± 0.03 |            |
| ILNi    |            |            | 15.70 ± 0.02 |

Table 2. Content of oxides in initial illite product IL

| Oxide | Content |
|-------|---------|
| SiO\(_2\) | 74.62 ± 0.16 |
| Al\(_2\)O\(_3\) | 14.65 ± 0.11 |
| Fe\(_2\)O\(_3\) | 1.15 ± 0.01 |
| CaO     | 4.10 ± 0.01  |
| MgO     | 3.71 ± 0.25  |
| SO\(_3\) | 0.87 ± 0.01  |
| K\(_2\)O | 0.54 ± 0.01  |
| TiO\(_2\) | 0.19 ± 0.01  |
| MnO     | 0.05 ± 0.01  |

Specific surface area of initial illite product IL and its modified Co\(^{2+}\), Cu\(^{2+}\) and Ni\(^{2+}\) forms – ILCo, ILCu and ILNi (Table 3) was determine according to STSA method. Samples of illite modified by cobalt and cooper contained micropores.

| Sample  | STSA (m\(^2\)/g) |
|---------|------------------|
| ILCo    | 30.7             |
| ILCu    | 23.5             |
| ILNi    | 27.0             |
3.1. Thermal analysis

Illite product IL contains a certain amount of interlayer molecular water evaporated till 350 °C. Thermal decomposition of other samples starts by evaporating water molecules and molecules coordinating exchangeable cations in the interlayer in the range of 41 °C to 276 °C. Dehydration process corresponds to a first and second weight loss: IL 4.98 % (Figure 2a), ILCo 6.96 % (Figure 2b), ILCu 4.25 % and 5.26 % (Figure 2c) and ILNi 5.30 % (Figure 2d). Dehydration process fluently passes into dehydroxylation process between 400 °C and 880 °C where OH− groups are released from the structure. Final weight loss for commercial IL was 10.70 %, ILCo 13.43 %, ILCu 12.20 % and ILNi 9.71 %. Two DTG maxims were observed at samples ILCo at the temperature of 395 °C and weight loss of 3.20 % and second one at the temperature of 643 °C and weight loss of 3.27 % and ILCu at the temperature of 532 °C with the weight loss of 1.38 % and the second at the temperature of 748 °C with the weight loss of 1.31 %.

![Figure 2a](image1.png)
![Figure 2b](image2.png)
![Figure 2c](image3.png)
![Figure 2d](image4.png)

Figure 2. Thermal analysis of commercial illite product IL and its modified forms (ILCo, ILCu, ILNi).

3.2. Infrared spectroscopy

Infrared spectroscopy in the middle range between 4000 cm\(^{-1}\) – 400 cm\(^{-1}\) was used to characterize commercial illite and its modified monoionic forms in the aim to study their sorption properties and structural changes caused by the chemical modifications. Bands belonging mainly to valence vibrations and deformation vibrations of structural OH groups corresponding to the wavelength range 3750 cm\(^{-1}\) to 3400 cm\(^{-1}\) and 900 cm\(^{-1}\) to 600 cm\(^{-1}\) as well as valence vibrations and deformation vibrations of Si-O groups in the wavelength range from 1200 cm\(^{-1}\) to 700 cm\(^{-1}\) and 600 cm\(^{-1}\) – 150 cm\(^{-1}\) were determined. In the illite product IL and its modified forms ILCo, ILCu and ILNi are valence vibrations of OH groups, in the region of ~ 3611 cm\(^{-1}\) – 3616 cm\(^{-1}\), which are located at the edges of the illite plates. The deformation vibrations of the OH groups were absorbed at 911 cm\(^{-1}\) – 912 cm\(^{-1}\) (AlAlOH) and 836 cm\(^{-1}\) – 837 cm\(^{-1}\) (AlMgOH). The observed absorption bands at wavelengths of 3385 cm\(^{-1}\) – 3406 cm\(^{-1}\) belong to the valence vibrations of the adsorbed water of the H-O-H groups. The visible absorption band at wavelengths of 1629 cm\(^{-1}\) and 1632 cm\(^{-1}\) belongs to the deformation vibrations of the adsorbed water of H-O-H groups.
The commercial product illite IL (Figure 3a) as well as its modified forms ILCo (Figure 3b) and ILNi (Figure 3d) have in the infrared spectrum an absorption band present at 1426 cm\(^{-1}\) - 1436 cm\(^{-1}\), belonging to the valence vibrations of the CO\(_3^{2-}\) anions of the calcium carbonate. In the IR spectrum of ILCu (Figure 3c) are absorption bands present at 1550 cm\(^{-1}\) and 1425 cm\(^{-1}\), which may be related to the presence of copper acetate, respectively. copper complex.

The valence vibrations of the Si-O groups of the tetrahedral network have typical sign of aluminosilicates corresponding to the wavelength of 992 cm\(^{-1}\) – 996 cm\(^{-1}\). The deformation vibrations of Si-O-Al correspond to the absorption bands at ~ 511 cm\(^{-1}\) – 513 cm\(^{-1}\).

![Infrared spectra of commercial illite (IL) and its modified forms (ILCo, ILCu, ILNi).](image)

**Figure 3.** Infrared spectra of commercial illite (IL) and its modified forms (ILCo, ILCu, ILNi).

3.3. **X-ray diffraction powder analysis**

In the presented diffraction records of the IL product and in its modified forms ILCo, ILCu and ILNi (Figure 4), the mixed-layered mineral illite / smectite was identified as the main mineral according to the diffraction maximum. According to X-rays Diffraction maxima, which correspond to the admixture of quartz (Q) and feldspar (Fld), were also identified.

Since in the modified samples of ILCo, ILCu and ILNi is increasing the interplanar distance d (001) comparing to the illite product IL, we can assume that this increase was caused by the intercalation of Co\(^{2+}\), Cu\(^{2+}\) and Ni\(^{2+}\) ions into the interlayer space of smectite, in a mixed layer illite/smectite minerals. The mineral illite itself has no ability to swell.
Figure 4. Diffraction records of the IL product and its modified forms (ILCo, ILCu, ILNi).

3.4. Study of rheology and curing characteristics

The influence of the amount and selected types of used clay fillers on the course of sulfur vulcanization of tread mixtures (named: IL, ILCo, ILCu and ILNi) was evaluated on the basis of selected parameters of vulcanization characteristics from rheological records of vulcanization curves of given mixtures. The following vulcanization characteristics of rubber blends were evaluated: minimum torque $M_L$ and maximum $M_H$ torque, onset $t_s$, optimal crimping time $t_{c(90)}$ and curing rate coefficient $R_v$. The course of individual vulcanization curves of tread rubber compounds with the content of selected fillers based on clay products is almost comparable with the course of the vulcanization curve of the comparative standard mixture ST.

3.4.1. Evaluation of minimal and maximal torque. Minimal torque values $M_L$ (Figure 5) of samples ILCo and ILNi containing 1 phr of alternative modified filler were slightly higher (1.79 dN.m) than the standard ST 1.73 dN.m. Higher values were observed at the samples ILCo and ILCu with the filler content of 8 phr and ILCu with 1 phr filler content. Lower values $M_L$ were observed at the samples containing commercial illite product and ILNi with a filler content of 8 phr. The lowest value was visible at the sample IL 1 phr of 1.55 dN.m.

Maximal torque values $M_H$ (Figure 6) of all samples rubber blends with contain clay fillers IL, ILCo, ILCu or ILNi were lower than the standard ST value (12.33 dN.m). Slightly difference was observed at the samples ILCu with 1 phr filler and ILNi with 1 phr of filler. The lowest maximal torque was obtained at the sample with a content of illite product 8 phr IL (10.67 dN.m).

Figure 5. Minimal torque $M_L$.

Figure 6. Maximal torque $M_H$. 
3.4.2. **Evaluation of curing characteristics.** Safety processing $t_s$ (Figure 7) value of standard ST was comparable with the sample named ILCo with a content of 1 phr filler. Other samples obtained higher values $t_s$. The highest value $t_s$ was achieved by a sample ILCo with a content of 8 phr filler. On the other hand, lowest value $t_s$ was observed by a ILCu at 1 phr of filler.

Optimal curing time $t_{c(90)}$ (Figure 8) of standard ST 5.77 min was comparable with the sample ILCu 1 phr 5.75 min. Similar value $t_{c(90)}$ but little bit lower was reached by a sample ILCo with 1 phr of modified filler and IL commercial product with 1 phr of a filler. Higher value $t_{c(90)}$ were observed at the samples IL 8 phr, ILCo 8 phr and ILNi both 1 phr and 8 phr. The highest optimal curing time was observed at the sample ILCu with 8 phr of modified filler for 9.40 min.

![Figure 7. Safety processing.](image7)

![Figure 8. Optimal curing time.](image8)

Curing rate coefficient $R_v$ (Figure 9) of sample ILNi 24.75 min$^{-1}$ with 8 phr was comparable to a standard ST 24.69 min$^{-1}$. The lowest value was achieved by a sample ILCu with 8 phr of modified filler of 14.73 min$^{-1}$. Lower curing rate coefficients were observed at the samples IL commercial illite at 8 phr, ILCo of 22.27 min$^{-1}$ and 8 phr of modified filler, then ILCu 23.04 min$^{-1}$. Higher coefficients $R_v$ were achieved by a samples ILCo with 1 phr of modified filler at the 26.39 min$^{-1}$ and ILNi 25.32 min$^{-1}$ at the 1 phr of modified filler. The highest value $R_v$ (29.07 min$^{-1}$) was observed at the sample IL with containing filler 1 phr.

![Figure 9. Curing rate coefficient.](image9)

3.5. **Study of physical and mechanical properties**

The influence of the amount and selected types of used clay fillers on the physical and mechanical properties: tensile strength, elongation at break and hardness was evaluated for prepared tread vulcanizates containing selected fillers based on clay products before and after accelerated thermal aging. All prepared tread vulcanizates containing selected fillers (ILCo, ILCu or ILNi) based on illite products were compared with a standard ST vulcanizate with containing carbon black N339.
3.5.1. Evaluation of tensile strength before and after accelerated thermal aging

All tensile strength values (Figure 10) before accelerated thermal aging were higher than standard (17.76 MPa) but only ILCo with 8 phr of modified filler was lower 17.50 MPa. The highest values were achieved by a ILNi at 8 phr for 19.46 MPa, then it was 19.19 MPa IL Cu of 8 phr modified filler, commercial illite in the amount of 1 phr for 19.08 MPa. After accelerated thermal aging in air for standard ST observed 17.63 MPa. Other values were higher than the standard but the one commercial illite IL at 1 phr was lower 16.16 MPa.

![Figure 10. Tensile strength before and after accelerated thermal aging.](image)

3.5.2. Evaluation of elongation at break before and after accelerated thermal aging

Elongation at break (Figure 11) before accelerated thermal aging of all samples containing modified fillers and commercial illite obtained higher values than the standard ST 340.51 %. The highest value was observed by a ILNi with 8 phr of filler 453.49 %. All samples containing 8 phr of modified filler obtained higher values of elongation at break. Samples with an amount of modified filler of 1 phr were higher than a standard ST. The highest value was obtained by a sample IL 1 phr 381.28 %. Standard’s elongation at break after accelerated thermal aging was 266.38 %. Slightly higher values were achieved by all samples containing 1 phr of modified filler after accelerated thermal aging, all samples with 8 phr content of modified filler were higher than before aging. The highest value was observed at the sample ILNi 8 phr 337.11 %.

![Figure 11. Elongation at break before and after accelerated thermal aging.](image)
3.5.3. **Evaluation of hardness before and after accelerated thermal aging.** Comparable values of hardness (Figure 12) before accelerated thermal aging were reached by a sample IL Cu with a content of modified filler 1 phr 60.8 Shore A and IL Ni with 1 phr modified filler 60.5 Shore A. Other values were a bit lower but still similar to standard 60.4 Shore A. After accelerated thermal aging were comparable values of samples commercial illite at 8 phr content and IL Co with 1 phr batched filler. Lower values were observed for samples IL 63.4 Shore A, 63.9 Shore A IL Co with 8 phr of filler, IL Cu 63.4 Shore A with 8 phr of filler and IL Ni 62.8 Shore A. Highest value was obtained by a sample IL Ni 67.3 Shore A with a 1 phr content of modified filler.

![Figure 12. Hardness before and after accelerated thermal aging.](image)

3.5.4. **Rebound resilience.** Rebound resilience test was performed for samples with an 8 phr content of modified filler. Rebound resilience (Figure 13) was higher for all samples with modified fillers. The highest value was reached by a sample IL Ni. The higher rebound, the lower rolling resistance is.

![Figure 13. Rebound resilience results of 8 phr batched modified fillers (ILCo, ILCu and ILNi).](image)

4. **Conclusion**

The submitted work was focused on preparation and characterization of modified monoionic forms of illite product and also of initial illite product. Modified forms (ILCo, ILCu and ILNi) were prepared form layered aluminosilicates by a change of cations Co$^{2+}$, Cu$^{2+}$ and Ni$^{2+}$. Prepared modified samples were subjected to a spectral analysis by an infrared spectroscopy, x-ray fluorescence energy-dispersive
spectroscopy, x-ray diffraction analysis and thermal analysis by a thermogravimetry and differential thermogravimetry and also their specific surface area was determined.

The processes of dehydration and dehydroxylation of the commercial products and their monoionic forms were monitored by thermal analysis with appropriate changes on the DTG curves and the weight changes were evaluated from the TG curves.

Thermal decomposition in the commercial product IL and its modified forms ILCo, ILCu and ILNi begins with the dehydration process. The weight loss of H₂O adsorbed on the surface and in the interlayer space of illite products has the following order: IL <ILNi <ILCo <ILCu. The dehydration process passes smoothly into the dehydroxylation process in the temperature range of about 400 °C to 880 °C and hydroxyl OH groups begin to be released from the structure.

The results of infrared spectroscopy show that the structural modifications of tetrahedral and octahedral networks caused by adsorbed Co²⁺, Cu²⁺ and Ni²⁺ cations had an effect on the valence and deformation vibrations of OH and Si-O groups.

The valence vibrations and deformation vibrations of OH and Si-O groups are visible in the illite product IL and its modified forms (ILCo, ILCu and ILNi).

In the presented diffraction records of the IL product and in its modified forms, the mixed layer illite/smectite was identified as the main mineral. According to x-rays diffraction maximum corresponding to the mixture of quartz (Q) and feldspar (Fld) in all commercial products and their modified forms were also identified. Since the interstitial distance d (001) increases in the modified samples compared to the illite product IL, we can assume that this increase was caused by the intercalation of Co²⁺, Cu²⁺ and Ni²⁺ ions into the interlayer space of smectite, in the mixed layer of illite/smectite mineral. The mineral illite itself has no ability to swell.

The second part of the work dealt with the application of clay products: a commercial illite product and an modified illite products into a real tread rubber blend in the function of a filler as a partial replacement of a standardly used filler - carbon black N339. The vulcanization characteristics of the tread mixtures were studied and the physical-mechanical properties, accelerated thermal aging and rebound were studied for the prepared vulcanizates.

From the above results of rheology characteristics can be seen the decrease of minimal torque and maximal torque for every sample with a content of modified filler both 1 phr and 8 phr. On the other hand, curing characteristics obtained higher values for both processing safety and optimal curing time than the standard ST.

All tensile strength values before accelerated thermal aging were higher than standard but only ILCo with 8 phr of modified filler obtained lower value. After accelerated thermal aging in air were values of tensile strength higher than the standard ST but the one commercial illite IL at 1 phr was lower.

Elongation at break results showed the increase of values for samples containing 8 phr of modified filler before thermal aging in air, after accelerated thermal aging elongation at break results were also higher than the standard. In the case of 1 phr content same thing applies but commercial IL was the one with lower value.

A bit lower values of hardness for samples with a content of modified filler were achieved. The smallest values were observed in the commercial illite product for both 1 phr and 8 phr, but also for ILCu and ILNi at 8 phr of filler. After thermal aging were obtained higher values than standard.

Based on the obtained results we can state that modified commercial clay products fulfill the function of a filler and can be used as a partial replacement for a standardly used filler.

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