Experimental thermal transport evolution of silane activated nano-clay reinforced styrene butadiene elastomeric nanocomposites

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Abstract. In this study, silane activated nanoclay was reinforced in styrene butadiene rubber (SBR) to enhance the thermal resistance/stability and mechanical properties of SBR. Silane activated nanoclay with variant concentrations was impregnated in the rubber matrix to fabricate polymer nanocomposites under control processing conditions. Experimental thermal transport, thermal oxidation, phase transition study, and mechanical properties of the nanocomposite specimens were carried out. Thermal insulation, thermal stability, and heat flow response were remarkably enhanced with the addition of nanokaolinite in the polymer matrix. Phase transition temperatures, their corresponding enthalpies, tensile strength, elastic modulus, elongation at break and hardness of the rubber composites were positively influenced with the filler incorporation into the host matrix. The even dispersion of nanoreinforcements, morphological and compositional analyses of the thermal transport tested specimens were performed using scanning electron microscopy and energy dispersive spectroscopy, respectively.

1. Introduction
The clays study has become a prominent topic of the present era due to their easy and abundant availability, low cost, and environment friendly nature. These clays have multidisciplinary applications, i.e., construction, polymer, medicine, ceramic, and metal processing industries [1-3]. Montmorillonite, kaolinite, bentonite, etc were used in polymer matrices to enhance their thermal, physical, mechanical and fire retardant characteristics. Particle size of clay is an important parameter that influences the thermomechanical character of the base polymer. Nanoclays have several advantages over micro size clays due to their large surface area, nanoscale interaction with the polymeric chains and low density [4-6]. Kaolinite is an aluminosilicate mineral clay having layered structure used in paints, medicines, rubbers, ceramics, etc. Nanokaolinite remarkably influences the physical and chemical characteristics of the base polymer matrix. Filler-matrix compatibility and its uniform dispersion within the host polymer are the major reinforcing parameters that drastically change the characteristics of the fabricated composites. Functionalization of the incorporated nanofillers by using appropriate chemical method is one of the procedure viz. efficiently used in academia and industry to enhance filler to matrix compatibility. Silane functionalization route in
which adequate silane coupling agent is used to attach silane moiety is effective, easy, economical and environment friendly method to functionalize the incorporated reinforcements compared to other established chemical processes. Nanokaolinite (NK) with low density, high aspect ratio, large surface area and soft nature has affirmed the promising reinforcing effects if uniformly dispersed in the rubber matrices, i.e., ethylene propylene diene monomer rubber, styrene butadiene rubber (SBR), acrylonitrile butadiene rubber, neoprene rubber, etc [7-10]. SBR is a synthetic rubber, synthesized through emulsion or solution polymerization. It has good abrasion, aging and impact strength. The main application area of this rubber is tire, shoes and gasket industries. Usually carbon black is reinforced in the SBR rubber to attain the required level of mechanical strength but thermal resistance suffers with the incorporation of this filler [11-14]. During the tire operation, enormous heat is generated due to road friction which may degrade the mechanical characteristics of the rubber [15-18]. So, the filler that improves thermal endurance, thermal resistance and mechanical strength, synchronously is required for SBR to perform better during its usage [19].

Thermal conductivity, thermal impedance, thermal degradation, differential thermal analysis, differential scanning calorimetric study and mechanical properties are evaluated for NK/SBR composites in this novel research. In addition, the effect of nanokaolinite loadings on the glass transition, crystallization, melting temperatures and their associated specific enthalpies are also executed herein.

2. Experimental

2.1. Materials

Emulsified Styrene butadiene rubber (INDOPOL 1502) was bought from Evergreen Global Pte. Singapore. Silane coupling agent (SCA), 3-aminopropyl-trimethoxysilane (APTMS) and cetrimonium bromide (CTAB) were purchased from Sigma Aldrich, Germany. Kaolinite, nanosilica, sulphur, zinc oxide and stearic acid were purchased from Merck, Germany. Aromatic oil and wax were received from International Petrochemicals (Pvt) Ltd, Pakistan. Mercaptobenzthiazole Disulphide (MBTS) and Cyclohexyl Benzthiazyl Sulphenamide (HBS) were supplied by Dalian Richon Chemical Co. Ltd, China.

2.2. Synthesis of nanokaolinite with silane coupling agent

A silane coupling agent (SCA), 3-aminopropyl-trimethoxysilane (APTMS) along with cetrimonium bromide (CTAB) was used for the surface treatment to modify the layered silicate mineral clay (kaolinite). A specific amount of the unmodified kaolinite was added in ultra pure water in a ratio 10:100 and the mixture was sonicated in ultra-sonication bath at 70°C and 40 KHz frequency for 30min. After that, 1mass% CTAB was introduced in the preset mixture followed by further sonication under similar conditions for next 30min. Subsequently, 5 volume % SCA was added in the mixture and sonicated it for next three hours. Consequently, magnetic stirring followed at 1000rpm for an hour with 90°C. The sonicated solution was diluted five times to maintain its pH at 7. Centrifuge (Centurion Scientific C2 series) at 4000rpm was used to recover the nano alumino-silicate clay. The silane functionalized nanokaolinite (NK) sample was dried for 12h at 80°C to eradicate moisture from the specimen.

2.3 Fabrication of NK/SBR nanocomposites

A preset amount of nano-reinforcement (nanosilica), lubricant (aromatic oil + wax), surface modifier for nanoclay (stearic acid) and four progressive loadings (0–15wt %) of NK were inserted in the dispersion kneader chamber along with the SBR matrix at 110°C for 30 minutes to disperse nanofillers properly in the host matrix. Cross linker (sulphur), primary accelerator (MBTS), secondary accelerator (HBS), and activator (zinc oxide) were incorporated into the rubber matrix in the post mixing stage on a two roller mixing mill at 60°C and 30rpm roller’s speed for twenty minutes to disperse the reinforcements and processing aids homogeneously within the polymer matrix [20]. Table 1 illustrates the basic composition of SBR composite (SK1). The four silane functionalized nanoclay
concentrations, i.e., 0, 5, 10, and 15 wt% were impregnated in the rubber matrix and the four rubber nano compositions are nominated as SK1, SK2, SK3, and SK4, correspondingly. The base formulation of the polymer nanocomposite is SK1 and is illustrated in Table 1.

| SBR (wt%) | Nano Silica (wt%) | ZnO (wt%) | Stearic Acid (wt%) | Aromatic Oil (wt%) | Sulphur (wt%) | bHBS (wt%) | bMBTS (wt%) | Wax (wt%) |
|-----------|------------------|-----------|-------------------|-------------------|-------------|-----------|-----------|----------|
| 100       | 40               | 5         | 2.5               | 10                | 2.5         | 2         | 2.5       | 2.5      |

*a HBS: Cyclohexyl Benzthiazyl Sulphenamide
*bMBTS: Mercaptobenzthiazole Disulphide

2.1. Characterization

Scanning electron microscopy (SEM, JSM 6940A, Jeol, Japan) along with the energy dispersive spectroscopy was used to characterize the even dispersion of nanoclay, surface morphology of the post thermal conductivity/impedance tested nanocomposite specimens and their composition analysis. Thermal conductivity ($\lambda_{SN}$) of the polymer nanocomposite specimens were carried out using ASTM E1225-99. Experimental setup design to evaluate $\lambda_{SN}$ of the composite specimens in the temperature range 323K to 523K is depicted in Figure 1.

Schematic illustration of the comparative guarded longitudinal heat flow system shows the feasible locations of K-type thermocouples, heating source, water heat sink, temperature data logger, copper upper and lower meter bars and laptop [21]. Time–temperature profiles of all thermocouples located at specific positions were displayed on the laptop display using Squirrel OQ610 series temperature data logger. Thermal conductivity of the composite specimens was measured at different input temperatures using equation 1.

Thermal conductivity of specimen =$\lambda_{SN}= ((Q'_T + Q'_B)(d_4 -d_3)/2(v_4 - v_3))$  

Heat flow at the top bar = $Q'_T = \lambda_c * (v_2 - v_1)/(d_2 -d_1)$

Heat flow at the bottom bar = $Q'_B = \lambda_c * (v_6 - v_3)/(d_6 -d_3)$
\( v_1, v_2, v_3, v_4, v_5, \) and \( v_6 \) are the temperatures of the six thermocouples and \( d_1, d_2, d_3, d_4, d_5, \) and \( d_6 \) are their positions. \( \lambda_c \) is thermal conductivity of the copper bar. Thermal impedance of a material is its ability to resist thermal/temperature fluctuations in a variable heating environment. For thermal impedance four thermocouples have been used instead of six in the mentioned thermal conductivity design. Thermal impedance of the nanocomposite specimens were measured according to the ASTM D5470-03 and by following equation 2.

\[
\text{Thermal impedance (m}^2\text{-K/W)} \ = \ 4.09 = \ \lambda_c \ (V_A - V_D) \times (A/\Omega)
\]

\[
\Omega = V \ (\text{Voltage}) \times I \ (\text{Current})
\]

Temperature of upper bar surface in contact with the sample (K) = \( V_a = v_2 - ((v_1 - v_2) \times (d_b/d_a)) \)
Temperature of lower bar surface in contact with the sample (K) = \( V_a = v_3 - ((v_3 - v_4) \times (d_c/d_c)) \)

\( v_1 \) (K) = Upper temperature of upper bar \( v_2 \) (K) = lower temperature of upper bar
\( v_3 \) (K) = Upper temperature of lower bar \( v_4 \) (K) = Lower temperature of lower bar
\( d_a \) (m) = Distance between \( T_1 \) to \( T_2 \) \( d_b \) (m) = Distance between \( T_2 \) to \( S \) Sample
\( d_c \) (m) = Distance between \( T_3 \) to \( T_4 \) \( d_d \) (m) = Distance between \( S \) Sample to \( T_3 \)

Thermal degradation and endothermic/exothermic capability of the rubber nanocomposites were carried out using Perkin Elmer Diamond TG/DTA, Japan. Heating rate and temperature range during the thermogravimetric and differential thermal analyses was 10°C/min and 25-850°C, respectively. Perkin Elmer Diamond DSC, Japan was used to analyze the heat flow response of the polymer composites from -75°C-450°C to determine physical transformations such as phase transitions (glass, crystallization, and melting), and their specific enthalpies with a heating rate (10°C/minute) of polymer nanocomposites. Both of these studies were performed in the air atmosphere. Tensile strength, elongation at break, and modulus of elasticity of the polymer nanocomposite specimens were executed using universal tensile testing machine (AG-20KNXD Plus, Shimadzu) according to the ASTM standard D412-98A. Shore A hardness of polymer nanocomposites were measured from Torsee, Tokyo testing machine. Scanning electron microscopy (SEM, JSM 6940A, Jeol, Japan) along with the energy dispersive spectroscopy was used to analyze the surface morphology of the post thermal conductivity tested specimens and compositional analysis of the rubber nanocomposite. Fourier Transformation In-fared (FT-IR) spectroscopy in which Infrared spectrum measures the quantity of radiation absorbed as a function of its frequency. FT-IR (Perkin-Elmer Diamond 1000) was used to analyze the changes taken place on the nanokaolinite surface due to the silane treatment in the frequency range 400-4000 cm\(^{-1}\). 5200 Jeol Atomic force microscopy, Japan was used to examine the surface topography of the functionalized nanokaolinite.

3. Results and discussion

3.1. Confirmation of Silane functionalization

Figure 2 shows the FTIR spectra of unmodified/modified kaolinite. The peaks at 2922.91, 2849.14, and 1463.25 cm\(^{-1}\) identifies the presence of trimethoxy silane moiety at the surface of nanoakaolinite. Experimental results reveal that the functionalization of nanokaolinite due to the attachment of silane moiety in the presence of APTMS. The average particle size observed in Figure 3a is <1μm for the unmodified kaolinite. The synthesis and modification process has successfully reduced the particle size of the clay up to 1000 times. The average particle size noticed in Figure 3(b, c) for modified kaolinite is 15nm. Another interesting thing in the morphological analysis of the nanokaolinite is that all particles have approximately similar spherical shape and size. Another advantage of this route is that both the nano-particle synthesis and modification have taken place synchronously. The EDS analysis in Figure 3d, illustrates the presence of Al (21.05), Si (32.86), and O (46.09) present in nanokaolinite (Al\(_2\)Si\(_3\)O\(_9\)(OH)\(_4\)) that means synthesized nanoparticles maintain their original composition (unmodified kaolinite). AFM images clear the particle size fineness (1-5nm) of modified
nanokaolinite in Figure 4(a, b). The approximate average particle size observed in AFM and SEM images is less than 10nm. The measured average particle size of the fabricated modified nanokaolinite is around 5nm using SEM software. The morphology of the synthesized modified nanokaolinite is spherical.

Figure 2. FTIR Spectra of unmodified/modified kaolinite

Figure 3. Surface morphology of unmodified (a) and modified (b, c) kaolinite along with its compositional analysis (d)

Figure 4. Surface topography of modified Kaolinite

3.2. Thermal conductivity and thermal impedance analyses
Thermal conductivity contours of the rubber nanocomposites in the temperature range 323K to 523K are illustrated in Figure 5. Initially, thermal conduction through the SK1 composite is minimum compared to the clay nanocomposites. A drastic decline is observed in the $\lambda_{SN}$ values of the composite in the temperature range 333-373K, after that each rubber nanocomposite has established a separate level of $\lambda_{SN}$ in the proceeding TR. The SK4 nanocomposite having 15 wt% NK, has the least level of $\lambda_{SN}$ due to enormous heat quenching by the nanoclay during its phase changes and water evaporation. It elucidates that $\lambda_{SN}$ is diminished with the progressive addition of NK in the rubber matrix. The utmost NK loaded nanocomposite formulation has 41%, 22%, and 41% less $\lambda_{s}$ compared to 0 wt% concentrated SK1 composite specimen at 373, 423, and 473K, respectively due to the high surface area, excellent heat absorbing capability, and nanoscale interaction of NK with the polymer matrix[22-25].
Thermal impedance ($\zeta$) is an important characteristic of a polymer composite which ensures the capability of a material to resist thermal variation encounter during a specific application. Figure 6 simulates the effect of NK on the thermal impedance of the nanocomposite specimens. An elevation in $\zeta$ values up to 493K is observed in the graph and then a reduction is scrutinized in the further temperature line due to transpirational cooling effect associated with evaporation of aromatic oil within the nanocomposite specimens.

The nanocomposite SK4 has the maximum ability to resist incoming heat flux within the complete tested temperature range due to the maximum nanoclay incorporation into the polymer matrix [26, 27]. Figure 7 (a, b, c, and d) simulates the surface morphological analysis of the post thermal conductivity tested nanocomposite specimens in the temperature range 323-523K. A large number of micro voids are observed in SKI while the size and quantity of these pores are remarkably reduced with increasing NK concentration in the rubber matrix. It means that NK impregnated rubber nanocomposites has less surface rupture in the heating environment compared to the base polymer due to the excellent thermal stability and heat absorbing capability on the nanokaolinite. Figure 7e elaborates the compositional analysis of the 15 wt% loaded rubber composite. The elements C (59.99%), Al (5.69%), Si (12.55%), O (14.52%), S (1.10%), Ca (1.96%), Mg (2.58%) and Na (1.62%) are found in the energy dispersive spectroscopic analysis of the polymer nanocomposite.
3.3. Thermal decomposition and heat response of the polymer nanocomposites

Thermal endurance of the rubber nanocomposites were analyzed in air atmosphere from room temperature to 850°C and portrayed in Figure 8 and Table 2. An insignificant weight loss is observed up to 425°C and then the maximum thermal degradation is scrutinized in the proceeding 100°C due to polymer pyrolysis and polymeric molecular chain cession. From 550°C to 850°C, there is no significant change in weight loss is observed for the nanocomposite specimens due to the remaining carbon char and the nano inclusion.

A momentous enhancement in thermal endurance is noticed at all temperatures with the NK concentration elevation in the SBR matrix [28]. Figure 9 demonstrates the heat quenching potential of the NK/SBR nanocomposite. The composite specimens absorb enormous heat up to 300°C and the upward plateaus are observed up to 600°C due to the exhaust of volatile products included in the rubber nanocomposites.

Figure 7. SEM micrographs of the post thermal conductivity tested SC1 a), SC2 b), SC3 c), SC4 d) rubber nanocomposites and compositional analysis of 15 wt% loaded composite specimen.
Figure 8. Thermal degradation analysis of the nanokaolinite impregnated SBR nanocomposites in the temperature range 25°C – 850°C in air environment

Figure 9. Thermal absorbance capability of the rubber nanocomposites in the temperature range 25–850°C

| Table 2. Weight loss data of the nanocomposite specimens at various temperatures |
|---|---|---|---|---|
| Sample ID | Weight loss (%) at 200°C | Weight loss (%) at 400°C | Weight loss (%) at 600°C | Weight loss (%) at 800°C |
| SC1  | 1.274±0.01 | 3.271±0.01 | 65.843±0.01 | 69.887±0.01 |
| SC2  | 1.088±0.01 | 3.064±0.01 | 64.682±0.01 | 66.728±0.01 |
| SC3  | 0.259±0.01 | 2.591±0.01 | 63.432±0.01 | 66.064±0.01 |
| SC4  | 0.011±0.01 | 1.291±0.01 | 62.132±0.01 | 64.764±0.01 |

The heat absorbing capability of the nanocomposites is augmented with the progressive incorporation of NK in the rubber matrix due to the excellent thermal stability, high surface area, and nanoscale interaction of the silane functionalized nanokaolinite (NK) with polymeric molecular chains [29].

2.1. Differential scanning calorimetric study

Figure 10 illustrates the DSC analysis of the rubber nanocomposites in the temperature range -74°C to 450°C to analyze the effect of nanoclay concentration on the phase transition temperatures and their specific enthalpies. Glass transition temperature (T_g), crystallization temperature (T_c), onset melting temperature (T_m1), and offset melting temperature (T_m2) of the NK/SBR nanocomposites are compiled in Table 3. The T_g of the polymer nanocomposites is reduced with increasing filler concentration in the host matrix and a 10°C T_g reduction is noticed with the 15 wt% incorporation of NK in the rubber matrix. It means that the nano inclusion remarkably enhances the rubber phase of SBR nanocomposite by interacting with polymeric molecular chains at the nanoscale, reducing the crosslinking density of the base polymer, and due to the soft nature of the filler.

Crystallization temperature of the SN4 nanocomposite is more than the SK1 composite due to the excess NK loading in the former mentioned formulation. The progressive addition of the nanoclay has enhanced the T_c of the composite specimens due to the crystalline nature of the NK.

| Table 3. The measured data of the phase change temperatures of the NK impregnated rubber composites |
|---|---|---|
| Sample ID | T_g (°C) | T_c (°C) |
| SC1  | -74 | 155 |
| SC2  | -72 | 160 |
| SC3  | -71 | 162 |
| SC4  | -70 | 165 |
| Sample ID | Glass transition temperature ($T_g$, °C) | Crystallization temperature ($T_c$, °C) | Melting temperature ($T_{m1}$, °C) | Melting temperature ($T_{m2}$, °C) |
|-----------|-----------------------------------------|----------------------------------------|-----------------------------------|-----------------------------------|
| SC1       | -32.73±0.1                               | 76.23±0.1                              | 210.01±0.1                        | 372.05±0.1                        |
| SC2       | -38.94±0.1                               | 77.89±0.1                              | 209.58±0.1                        | 383.17±0.1                        |
| SC3       | -39.35±0.1                               | 78.88±0.1                              | 210.16±0.1                        | 388.65±0.1                        |
| SC4       | -42.70±0.1                               | 79.14±0.1                              | 211.60±0.1                        | 391.83±0.1                        |

The effect of the filler concentration on the melting characteristics of the rubber nanocomposite is opposite to $T_g$ and $T_c$. The melting temperatures $T_{m1}$ and $T_{m2}$ proceed in the following direction and the utmost wt% concentration of NK elevates $T_{m2}$ up to 19°C due to high thermal stability, heat absorbing capability, surface area and even dispersion of the nanoclay in the host polymer matrix [30-32]. The specific enthalpies of glass transition temperature and crystallization temperature phases are diminished while a significant evolution is noticed in the onset and offset melting phase transitions due to the additional heat absorbing capacity of the NK as tabulated in Table 4.

![Figure 10. Heat flow response of the nanocomposite specimens in the temperature range -74 to 450°C](image)

### Table 4. Specific enthalpies of the phase change temperatures of the polymer nano composites

| Sample ID | SC1           | SC2           | SC3           | SC4           |
|-----------|---------------|---------------|---------------|---------------|
| Specific Enthalpy of Glass transition (J/g) | 3.227±0.01    | 2.975±0.01    | 2.966±0.01    | 2.941±0.01    |
| Specific Enthalpy of Crystallization (J/g)  | 3.412±0.01    | 3.991±0.01    | 9.812±0.01    | 11.777±0.01   |
| Specific Enthalpy of First Melting Phase (J/g) | 4.369±0.01    | 11.073±0.01   | 21.605±0.01   | 37.191±0.01   |
| Specific Enthalpy of Second Melting Phase (J/g) | 182.610±0.01  | 204.756±0.01  | 220.499±0.01  | 257.184±0.01  |

The SK4 nanocomposite has 74 J/g more specific enthalpy compared to the SK1 formulation due to higher concentration of the reinforcing filler in the aforementioned composition compared to afterward, counterpart.

### 3.5. Mechanical properties

The stress–strain contours of the polymer nanocomposites are displayed in Figure 11, which simulates a remarkable evolution in toughness of the composite specimens with increasing filler incorporation into the rubber matrix. The effect of NK loading on the tensile strength, elongation at break, and...
modulus at 100% elasticity of the fabricated composite formulations is compiled in Figure 12a. The ultimate tensile strength is progressed from 2.78-5.10MPa, elongation at break is elevated from 137-161%, and modulus of elasticity at 100% elongation is promoted from 2.04-3.40Mpa with the 15 wt% addition of the silane functionalized nanokaolinite (NK) in the polymer matrix due to the high surface area, layered structure, even dispersion, and nanoscale interaction of the nanofiller with the polymeric molecular chains of the host matrix. Shore A hardness comparison of the rubber nanocomposites is displayed in Figure 12b [33-34].

Figure 11. Stress–strain contours of the nanoclay/SBR nanocomposites

Figure 12. The effect of nanokaolinite concentration on the ultimate tensile strength, elongation at break, and modulus 100% elasticity (a) and Shore A hardness (b) of the polymer nanocomposites with diverse nanoclay loadings

Shore A hardness is augmented from 46 to 51, 53, and 56 with the 5, 10, and 15 wt%, respectively progressive impregnation of nanokaolinite in the SBR base formulation (SK1) due to the enhancement in polymeric chain flow resistance offered by the silane functionalized nanokaolinite-SBR linkage at nanoscale.

4. Conclusion
Silane functionalized nanokaolinite, a layered silicate mineral is evenly dispersed in the styrene butadiene rubber matrix using dispersion kneader and two roller mixing mill. Thermal conductivity of the rubber nanocomposites is reduced up to 41%; thermal impedance is elevated up to 41%; thermal
stability is promoted up to 5%; glass transition is diminished up to 10°C; crystallization temperature, first and second melting phase temperatures are increased up to 3°C, 1.5°C and 19.7°C, correspondingly; ultimate tensile strength, rubber hardness, elongation at break, and modulus of elasticity at 100% elongation are augmented up to 2.37MPa, 10 Shore A, 24%, and 1.3MPa, respectively with 15 wt% nanoclay impregnation in the base composite formulation. Heat absorbing capability and toughness of the polymer nanocomposites are remarkably enhanced with the progressive incorporation of the nanofiller in the host rubber matrix. SEM analysis of the post thermal transport tested specimens reveals the micro void’s size and quantity reduction with increasing filler to matrix ratio.

5. Acknowledgements
The authors would like to greatly acknowledge Start Up Research Grant program (SRGP) from Higher Education Commission (HEC) of Pakistan (No: 21-433 SRGP/R&D/HEC/2014) for providing sufficient funds to execute this research; School of Chemical and Materials Engineering (SCME), National University of Sciences and Technology (NUST) Longman Mills, Lahore to facilitate us with the best of their rubber nanocomposite fabrication and testing expertise and facilities.

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