Experimental Techniques

The materials used for the study, the experimental techniques, and analytical methods employed for characterisation of the nanocomposites are discussed in this chapter.

2.1 Materials

2.1.1 Natural rubber

ISNR-5 grade of natural rubber with a Mooney viscosity (ML 1+4, 100 °C) 85 supplied by Rubber Research Institute of India, Kottayam was used for the study. Bureau of Indian Standards (BIS) specification for ISNR-5 is given in table 2.1 [1].

Table 2.1 BIS specifications of ISNR-5

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Parameters</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Dirt content, % by mass, max</td>
<td>0.05</td>
</tr>
<tr>
<td>2</td>
<td>Volatile matter, % by mass, max</td>
<td>0.8</td>
</tr>
<tr>
<td>3</td>
<td>Nitrogen, % by mass, max</td>
<td>0.6</td>
</tr>
<tr>
<td>4</td>
<td>Ash, % by mass, max</td>
<td>0.6</td>
</tr>
<tr>
<td>5</td>
<td>Initial plasticity, Po, Min</td>
<td>30</td>
</tr>
<tr>
<td>6</td>
<td>Plasticity retention index (PRI), min</td>
<td>60</td>
</tr>
</tbody>
</table>
Chapter 2

Natural rubber from the same lot has been used for the entire study since it is known that the molecular weight, molecular weight distribution and type and amount of non rubber constituents present are affected by clonal and seasonal variations, use of yield stimulants and method of preparation [2].

2.1.2 Natural rubber latex

Centrifuged natural rubber latex concentrate with minimum 60% dry rubber content (DRC), preserved with high ammonia (HA) preservative system and conforming to the BIS 5430-1981 specifications was used in the study. BIS specification for the high ammonia latex is given in table 2.2.

Table 2.2 BIS specifications for natural rubber latex (HA) concentrate

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Parameters</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Dry rubber content, % by mass min.</td>
<td>60.040</td>
</tr>
<tr>
<td>2</td>
<td>Total solids content, % by mass max.</td>
<td>61.050</td>
</tr>
<tr>
<td>3</td>
<td>Coagulum content, % by mass max.</td>
<td>0.030</td>
</tr>
<tr>
<td>4</td>
<td>Sludge content, % by mass max.</td>
<td>0.007</td>
</tr>
<tr>
<td>5</td>
<td>Alkalinity as ammonia, % by mass</td>
<td>0.730</td>
</tr>
<tr>
<td>6</td>
<td>KOH number</td>
<td>0.496</td>
</tr>
</tbody>
</table>

2.1.3 Acrylonitrile butadiene rubber (NBR)

Nitrile Rubber (N-03868) with acrylonitrile content 40 weight percentage supplied by M/s Gujarat Apar polymers Ltd, Mumbai, India was used for the study.
Experimental Techniques

2.1.4 Nanoclay

Montmorillonite clays used in this study were provided by Southern clay Products Inc., USA. Unmodified clay used was sodium montmorillonite clay (cloisite Na⁺), which has an ion exchange capacity of 92.6meq/100g clay and an interlayer distance of 11.7Å. Two different types of organically modified nanoclays differing in the interlayer distance and in modifying groups were also used for this study. The interlayer distance of the clays used were 18.3Å, and 30.6Å for cloisite 10A [2MBHT: dimethyl, benzyl, hydrogenated tallow, quaternary ammonium], cloisite 15A [2M2HT: dimethyl, dihydrogenated tallow, quaternary ammonium] respectively.

2.1.4.1 Cloisite 10A

Quaternary ammonium salt with the following structure is used as the modifier for cloisite 10A clay

\[
\text{CH}_3
\]

\[
\text{CH}_3 - \text{N}^+ - \text{CH}_2 - \text{C}_6\text{H}_5
\]

\[
\text{HT}
\]

HT : Hydrogenated Tallow (~65% C18, ~30% C16, ~5% C14)

Anion : Chloride

2MBHT: dimethyl, benzyl, hydrogenated tallow, quaternary ammonium

2.1.4.2 Cloisite 15A

Quaternary ammonium salt with the following structure is used as the modifier for cloisite 15A clay
Chapter 2

\[
\begin{align*}
\text{CH}_3 \\
\mid \\
\text{CH}_3 - \text{N}^+ - \text{HT} \\
\mid \\
\text{HT}
\end{align*}
\]

HT: Hydrogenated Tallow (~65% C18, ~30% C16, ~5% C14)

Anion: Chloride

2M2HT: dimethyl, dihydrogenated tallow, quaternary ammonium

Properties of nanoclay used for the study are given in table 2.3

**Table 2.3 Properties of nanoclays**

<table>
<thead>
<tr>
<th>Nanoclay</th>
<th>Organic modifier</th>
<th>Ion exchange capacity</th>
<th>% Moisture</th>
<th>% Weight Loss on ignition</th>
<th>Specific Gravity, g/cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cloisite 10A</td>
<td>2MBHT</td>
<td>125 meq/100g clay</td>
<td>&lt;2%</td>
<td>39%</td>
<td>1.90</td>
</tr>
<tr>
<td>Cloisite 15A</td>
<td>2M2HT</td>
<td>125 meq/100g clay</td>
<td>&lt;2%</td>
<td>43%</td>
<td>1.66</td>
</tr>
<tr>
<td>Cloisite Na'</td>
<td></td>
<td>92.6 meq/100g clay</td>
<td>&lt;2%</td>
<td>7%</td>
<td>2.86</td>
</tr>
</tbody>
</table>

46
2.1.5 Carbon black

High Abrasion Furnace black (N330), a product of M/s. Philips Carbon Chemicals Ltd Kochi was used. It had the following specifications

Table 2.4 Specifications for N330 (HAF) black

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Parameters</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Appearance</td>
<td>Black granules</td>
</tr>
<tr>
<td>2</td>
<td>DBP absorption (cc/100g)</td>
<td>102</td>
</tr>
<tr>
<td>3</td>
<td>Pour density (Kg/m³)</td>
<td>376</td>
</tr>
<tr>
<td>4</td>
<td>Iodine adsorption number</td>
<td>82</td>
</tr>
<tr>
<td>5</td>
<td>Loss on heating (%)</td>
<td>2.5</td>
</tr>
</tbody>
</table>

2.1.6 Coupling agent

The coupling agent used was Si69, a product of Degussa AG, Germany. Chemically Si69 is bis (triethoxysilyl propyl) tetrathiol and the molecular formula is \((\text{C}_2\text{H}_3\text{O})_3\text{Si}(\text{CH}_2)_3\text{S}_4\text{CH}_2)_3\text{Si}(\text{OC}_2\text{H}_5)\). The specification of Si69 is given in table 2.5 [3].
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Table 2.5 Properties of Silane coupling agent (Si69)

<table>
<thead>
<tr>
<th>Sl.No</th>
<th>Parameters</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Molecular weight</td>
<td>539.06</td>
</tr>
<tr>
<td>2</td>
<td>Sulphur content</td>
<td>22±1%</td>
</tr>
<tr>
<td>3</td>
<td>Viscosity at 25 °C</td>
<td>&lt;16.0 cps</td>
</tr>
<tr>
<td>4</td>
<td>Flash point</td>
<td>&gt;156 °C</td>
</tr>
<tr>
<td>5</td>
<td>Density at 25 °C</td>
<td>1.080-1.10</td>
</tr>
<tr>
<td>6</td>
<td>Refractive Index</td>
<td>&gt;1.480</td>
</tr>
</tbody>
</table>

2.1.7 Other chemicals

Compounding ingredients used for the study were zinc oxide (activator) supplied by M/s Meta Zinc Ltd. Mumbai; Stearic acid (co-activator) supplied by Godrej Soap (P) Ltd. Mumbai; N-Cyclohexyl-2-Benzothiazole Sulphenamide (CBS) (accelerator) of the class I sulphenamide supplied by Polyolefins Industries Ltd. Mumbai and N (1,3-dimethylbutyl) N-phenyl-p- phenylene diamine (anti-oxidant) called Vulkanox PPD supplied by Bayer India Ltd. Mumbai. Quaternary ammonium surfactants viz, Cetyl trimethyl ammonium bromide (CTAB), Cetyl pyridinium chloride (CPC), Benzalkonium chloride (BAC) used for the organophilic modification of the clay were obtained from Loba chemie Pvt. Ltd, Mumbai.

Toluene and Methyl ethyl ketone used in the present investigation were of analytical grade. Methyl methacrylate (MMA) used for the preparation of MMA grafted NR was obtained from Loba chemie Pvt. Ltd, Mumbai.
2.2 Experimental methods

2.2.1 Characterisation techniques

2.2.1.1 Characterisation using X-ray diffraction technique

X-ray diffraction (XRD) was used to study the nature and extent of dispersion of the clay in the composite. XRD were collected using Bruker, D8 Advance diffractometer at the wavelength CuKα = 1.54 Å, a tube voltage of 40 KV and tube current of 25 mA. Bragg’s law defined as \( n\lambda = 2d\sin\theta \), was used to compute the crystallographic spacing (d) for the clay. The samples were scanned in step mode by 1.0°/min, scan rate in the range of 2 to 12°.

2.2.1.2 Scanning electron microscopy (SEM)

In SEM, the electron beam incident on the specimen surface causes various phenomena of which the emission of secondary electrons is used for the surface analysis. Emitted electron strikes the collector and the resulting current is amplified and used to modulate the brightness of the cathode ray tube. There is a one to one correspondence between the number of secondary electrons collected from any particular point on the specimen surface and the brightness of the analogous point on the screen and thus an image of the surface is progressively built up on the screen. Scanning electron microscopic studies of the vulcanisates were carried out on the freshly cut surface in a Hitachi SE Microscope (model H 6010). The sample surface was gold coated prior to the examination.

2.2.1.3 Transmission electron microscopy

The transmission electron microscopy was performed using a JEOL JEM - 2010 (Japan) transmission electron microscope, operating at an accelerating voltage of 200 KV. The samples for TEM analysis were prepared by ultra-cryomicrotomy using a Leica Ultracut UCT. Freshly sharpened glass knives with cutting edge of 45° were used to get the cryosections of 50 - 70 nm thickness. Since these samples were elastomeric in nature, the temperature during ultra cryomicrotomy was kept at -70 °C.
The cryosections were collected individually on sucrose solution and directly supported on a copper grid of 300-mesh size.

2.2.2 Preparation of rubber clay nanocomposites

The mixing was done on a laboratory size two roll-mixing mill (16 × 33 cm) at a friction ratio of 1:1.25 for natural rubber 1:1.1 for nitrile rubber as per ASTM D 3182-89. A nip gap of 0.2mm was set and the temperature maintained at 70 ± 5 °C. For the mixing of rubber with compounding ingredients, the rubber was passed through the rolls and it was banded over the front roll. After the nerve had disappeared, the compounding ingredients were added as per the procedure given in ASTM D 3184-89 (2001). After mixing, the stock was homogenised by passing through the tight nip of the mill for six times and sheeted out at a nip gap of 3 mm. Mixing time and temperature were controlled during the process.

2.2.3 Cure characteristics

Cure characteristics of the mixes were determined as per ASTM D 2084-1995 using Rubber Process Analyzer, RPA 2000®, which is a computer controlled torsional dynamic rheometer with a unique test gap design, an advanced temperature control system and fully automated operation modes. Testing environments employed in the present work are given in table 2.6.
To determine the cure characteristics of the rubber compound, approximately 5 g of the sample was placed in a sealed biconical cavity under pressurized conditions and submitted to harmonic torsional strain by the oscillation of the lower die through a small deformation angle of about $3^\circ$ and the transmitted torque is measured on the upper fixed wall [4]. The torque transducer on the upper die senses the force being transmitted through the rubber sample. A typical RPA cure curve is shown in the figure 2.1 and the following data can be obtained.

### Table 2.6 Testing parameters used for RPA

<table>
<thead>
<tr>
<th>Sl.No.</th>
<th>Parameters</th>
<th>Limit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Frequency</td>
<td>50 cpm</td>
</tr>
<tr>
<td>2</td>
<td>Strain angle</td>
<td>0.20 °</td>
</tr>
<tr>
<td>3</td>
<td>Temperature</td>
<td>150 °C</td>
</tr>
<tr>
<td>4</td>
<td>Time</td>
<td>30 min.</td>
</tr>
<tr>
<td>5</td>
<td>Die Type</td>
<td>Biconical</td>
</tr>
<tr>
<td>6</td>
<td>Die Gap</td>
<td>0.487 mm</td>
</tr>
<tr>
<td>7</td>
<td>Angle of oscillation of the Die</td>
<td>$3^\circ$</td>
</tr>
</tbody>
</table>
**Figure 2.1 Typical cure curve obtained from the Rubber Process Analyzer, RPA 2000**

Minimum torque, $M_L$

Torque obtained by mix after homogenising at the test temperature and before the onset of cure.

Maximum torque, $M_H$

Maximum torque recorded after the curing of the mix is completed.

Torque, $M_T$

Torque at any given time $T$

Optimum Cure time, $T_{90}$

This is the time taken for obtaining 90% of the maximum torque

Scorch time, $T_{10}$
Time for attaining 10% of the maximum torque

Induction time, $T_5$

It is the time taken for about 5% vulcanisation.

Cure rate index

Cure rate index is calculated from the following equation.

\[ \text{Cure rate index} = \frac{100}{T_{90} - T_{10}} \]

Where, $T_{90}$ and $T_{10}$ are the times corresponding to the optimum cure and scorch respectively.

The computer software analyses the cure curve and prints out the data after each measurement.

2.2.4 Moulding

The test specimens for determining the physical properties were moulded in standard moulds by compression moulding on an electrically heated hydraulic press having 45 cm x 45 cm platen at a pressure of 200 Kg cm$^2$. The rubber compounds were vulcanized up to their respective optimum cure time at 150 °C. Mouldings were cooled quickly in water at the end of the curing cycle and stored in a cool and dark place for 24 hours and were used for subsequent physical tests. Additional curing time based on the sample thickness for samples having thickness higher than 6 mm (hardness) was given to obtain satisfactory cure for the entire thickness of the sample.

2.2.5 Physical testing

Physical tests such as tensile strength, tear strength, hardness, etc have been carried out on vulcanised rubber to standardize the rubber compounds used in the rubber industry to meet various service requirements and to investigate product or process failures. Physical tests are also utilized for the quality assurance and the quicker prediction of quality of the prepared rubber products. For each tests at least six specimens were tested and the mean values are reported.
2.2.5.1 Tensile properties

The tensile strength is defined as the force per unit area of cross section, which is required to break the test specimen, the condition being such that the stress is substantially uniform over whole of the cross section. The cure conditions giving the highest tensile strength are being widely adopted as optimum cure. The elongation at break (EB) is the maximum value of elongation expressed as a percentage of the original length. The value of tensile stress (force/unit area) required to stretch the test piece from the unstrained condition to a given elongation is called modulus or more accurately tensile stress at a given strain.

The tensile properties of the vulcanisates were determined on a ‘Shimadzu Autograph AG-I series’ universal testing machine (UTM), using a crosshead speed of 500 mm/min as per ASTM D 412-1998 (Method A). All the tests were carried out at 28 ± 2°C. Dumbbell specimens for the test were punched out of the moulded sheet along the mill grain direction using a dumb bell die (C-type). The thickness of the narrow portion was measured using a digital thickness gauge. The sample was held tight by the two grips or jaws of the UTM, the upper grip being fixed. The tensile strength, elongation at break and modulus were evaluated and printed out after each measurement by the microprocessor.

2.2.5.2 Tear resistance

This test was carried out as per ASTM D 624 -1998 using unnicked, 90° angle test pieces. The samples were cut from the compression-moulded sheets parallel to the mill grain direction. The test was carried out on the Shimadzu Autograph AG-I series universal testing machine (UTM). The speed of extension was 500 mm/min and the test temperature was 28 ± 2°C. The thickness of the narrow portion was measured using a digital thickness gauge. The sample was held tight by the two grips or jaws of the UTM, the upper grip being fixed. The tear resistance of the samples was reported in N/mm.
2.2.5.3 Hardness

The hardness (Shore A) of the moulded samples was determined using Zwick 3114 hardness tester in accordance with ASTM D 2240 -1997. The tests were performed on unstressed samples of 30 mm diameter and 6 mm thickness. Readings were taken after 15 seconds of the indentation after a firm contact has been established with the specimens.

2.2.5.4 Heat build up

The Ektron flexometer conforming to ASTM D 623-1999 was used for measuring the heat build-up. A cylindrical sample of 25 mm in height and 19 mm in diameter was used for the test. The oven temperature was maintained at 100 °C. The samples were placed in the preconditioning oven for 20 minutes at 100 °C. After preconditioning the test samples were subjected to a flexing stroke of 4.45 mm under a load of 10.9 kg and the temperature rise at the end of 20 minutes was taken as the heat build up.

2.2.5.5 Abrasion resistance

The abrasion resistances of the samples were measured with a DIN Abrader (DIN 53516). Moulded samples of 6 ± 0.2 mm diameter and 12 mm thickness were prepared as per ASTM D3183 and abrasion loss was measured as per ASTM D5963-04. The sample is kept on a rotating sample holder and 10 N load was applied. Initially a pre run is given for the sample and its weight taken. The weight after the final run is also noted. The difference in weight is the weight loss on abrasion. The volume loss on abrasion was calculated using the equation

\[
\text{Volume loss on abrasion} = \frac{\text{weight loss on abrasion}}{\text{specific gravity of the sample}}
\]

Abrasion resistance is the reciprocal of volume loss on abrasion.
Chapter 2

2.2.5.6 Strain sweep studies

The strain sweep measurements on green compound were conducted to study the rubber-filler interaction. Rubber Process Analyzer (RPA 2000-Alpha Technologies) is a purposely modified commercial dynamic rheometer [5]. Such instrument was modified for capturing strain and torque signals, through appropriate software. Filled rubber compounds exhibit strong non-linear viscoelastic behaviour, the well-known Payne effect, i.e. the reduction of elastic modulus with increasing strain amplitude [6]. RPA can do strain sweep tests in which the variation of storage modulus (G'), loss modulus (G'') and complex modulus (G*) with change in strain amplitude are measured. With respect to its measuring principle, the RPA cavity must be loaded with a volume excess of test material. In agreement with ASTM 5289, the manufacturer recommends to load samples of about 5.0 g (4.4 cm³) for a standard filled rubber compound with a specific gravity of 1.14 g/cm³. Samples for RPA testing were consequently prepared by die cutting 46 mm diameter disks out of around 2 mm thick sheets of materials. The testing temperature was selected as 100°C; a temperature below the curing temperature and the shear strain was varied from 0.5 to 100% keeping the frequency measurements at 0.5 Hz.

2.2.5.7 Dynamic mechanical analysis (DMA)

DMA works by applying an oscillating force to the material and the resultant displacement of the sample is measured. From this, the stiffness can be determined and tan δ can be calculated. Tan δ is the ratio of the loss component to the storage component. By measuring the phase lag in the displacement compared to the applied force it is possible to determine the damping properties of the material. Tan δ is plotted against temperature and glass transition is normally observed as a peak since the material will absorb energy as it passes through the glass transition. Dynamic mechanical thermal analysis (DMTA) spectra were recorded on rectangular specimens (length × width × thickness = 5 cm × 4 cm × 2.11 cm) in the tensile mode at a frequency of 1 Hz with a TRITEC2000B DMA 1L dewar (Triton-technology, U K).
DMTA spectra [i.e., the storage modulus and mechanical loss factor ($\tan \delta$)] were measured from -120 to 30 °C at a heating rate of 1°C/min.

2.2.6 Cross link density and swelling studies

Circular specimen of 20 mm diameter and 1mm thickness were cut using a sharp edged circular disc shaped die from the vulcanized rubber sheet. The samples were immersed in airtight test bottles containing about 20 ml of toluene maintained at constant temperature. Samples were removed periodically and the surface absorbed solvent drops were wiped off carefully by placing them between filter wraps. The mass of the sorbed sample was determined immediately on a digital balance with an accuracy of ±0.01mg. As the weighing was done within 30-40 seconds the error due to evaporation of other than surface adsorbed liquid is considered insignificant. The weighing of the sample is continued till the equilibrium swelling is attained. For calculating the crosslink density, desorbed weights of the samples were also taken after complete removal of the solvent.

The mole percent uptake of the sample is calculated from the diffusion data. The $Q_t$ values were determined as

$$Q_t = \frac{(\text{Wt. of the solvent sorbed at a given time})}{(\text{Mol. wt. of the solvent})} \times 100$$

At equilibrium swelling $Q_t$ becomes $Q_\infty$.

The mechanism of diffusion was investigated using the equation

$$\log \frac{Q_t}{Q_\infty} = \log k + n \log t$$

The value of $k$ depends on the structural features of polymer, whereas the value of $n$ determines diffusion mechanism. For the Fickian mode, case 1, the value of $n$ is 0.5 and it occurs when the rate of diffusion of penetrant molecules is much less than the relaxation rate of the polymer chains. For case 2 or non-Fickian transport, where the $n$ value is 1, the diffusion is rapid when compared with the simultaneous relaxation
process. However in the case of anomalous transport where the n value is in between 0.5 and 1, both solvent diffusion and polymer relaxation rate are comparable.

The effective diffusivity, $D$ of the rubber solvent system was calculated from the initial portion of the sorption curves using the equation [7, 8]

$$D = \pi \left[ \frac{h\theta}{4Q_{\infty}} \right]^2$$

Where $h$ is the initial thickness of rubber sample, $\theta$ the slope of the linear portion of the sorption curve $Q_t$ versus $t$.

The permeation of a solvent into a polymer membrane will also depend on the sorptivity of the penetrant in the membrane. Hence sorption coefficient $S$ has been calculated using the relation [9]

$$S = \frac{W_s}{W}$$

Where $W_s$ is the weight of the solvent at equilibrium swelling and $W$ the initial weight of the polymer sample.

Since the permeability depends on both diffusivity and sorptivity, the permeation coefficient has been determined using the relation [9]

$$P = D \times S$$

As diffusion is influenced by polymer morphology, the molar mass between cross links $M_c$ from the sorption data is also determined. The rubber solvent interaction parameter, which is needed for the estimation, has been calculated using the equation [9]

$$\chi = \beta + \frac{V}{RT} \left( \frac{\partial s}{\partial p} \right)^2$$
Where $V_s$ is the molar volume of the solvent and $p$ are solubility parameters of solvent and polymer taken from the polymer handbook. $R$ is the universal gas constant and $T$ the absolute temperature, $\beta$ is the lattice constant and is 0.38 in the calculation.

Using $\chi$ values the molar mass between crosslinks ($M_c$) of the polymer was estimated from the Flory Rehner equation [10, 11]

$$M_c = \frac{-\rho_p V_s (V_r)^{1/3} \chi}{\ln(1-V_r) + (V_r) + \chi (V_r)^2}$$

The volume fraction of rubber in the deswollen network was then calculated using the equation [12]

$$V_r = \frac{(d - fw) \rho_p^{-1}}{(d - fw) \rho_p^{-1} + A_o \rho_s^{-1}}$$

Where $d$ is the desorbed weight of the polymer, $f$ the weight percent of filler, $w$ the initial weight of the polymer, $\rho_p$ and $\rho_s$ the density of polymer and the solvent respectively and $A_o$ the net solvent uptake of the polymer. The monomeric molecular weight of NR being 68 g mol$^{-1}$, the average number of monomeric units between crosslinks can be calculated. A decrease in the value is observed. Using the $M_c$ values crosslink density can be calculated using the equation $v = 1/2M_c$

The expansion of the rubber in the presence of a solvent will significantly modify the conformational entropy ($\Delta S$) and the elastic Gibbs free energy ($\Delta G$). The elastic gibbs free energy can be determined from the Flory- Huggins equation [13]

$$\Delta G = RT \left[ \ln(1 - V_r) + (V_r) + \chi (V_r)^2 \right]$$
2.2.7 Gas permeability testing

The air permeability of the rubber vulcanisates were measured using Lyssy Manometric Gas Permeability Tester L100-2402. The test gas used was oxygen at a rate of 500ml/minute. Permeability of the samples is calculated using the equation, \( P_m = \frac{(T_s \times P_r)}{t_m} \), where \( P_m \) is the permeability of the test sample, \( t_m \) is the interval time constant for the test sample, \( P_r \) is the permeability of the reference (standard Polyethylene terephthalate (PET) sample) and \( T_s \) is the interval time constant for standard PET.

2.2.8 Infrared spectroscopy

Fourier transform infrared spectra are generated by the absorption of electromagnetic radiation in the frequency range 400 to 4000 cm\(^{-1}\) by organic molecules. Different functional groups and structural features in the molecules absorb energy at characteristic frequencies. The frequency and intensity of absorption are the indication of the bond strength and structural geometry in the molecule. The IR spectra of the modified clays and methacrylated natural rubber latex were recorded with Fourier Transform Infrared Spectroscope, Bruker, Tensor 27 model.

2.2.9 Thermal analysis

The thermogram of modified clays and rubber vulcanisates are recorded with a Thermo Gravimetric Analyzer Q-50, TA instruments. It is a computer-controlled instrument that permits the measurement of the weight changes in the sample material as a function of temperature. The sample placed in a temperature programmed furnace is subjected to temperature in the ranges 30 °C to 800 °C with a heating rate of 10 °C/minute and the corresponding weight changes was noted with the help of an ultra sensitive microbalance. Air and nitrogen were used as purge gases.
Experimental Techniques

References


3. Degussa, Product Information Sheet on Ultrasil VN3, PI 203. IE- from the website www.degussa-fp.com


