

**STUDIES ON THE MECHANICAL PROPERTIES OF BLENDS OF
NATURAL RUBBER AND CHLOROPRENE RUBBER**

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CHAPTER 1

1.0 INTRODUCTION

1.1 POLYMER BLENDING

Polymer blending has been described to be similar as alloying of metals. The properties of resulting blends depends upon a number of factors including the choice of parent polymers, compatibilizers, blend composition, moisture content and the method of blend preparation (Dyson, 1990). Compatibility of the parent polymers is an important issue, as polymers form a homogeneous mixture phase that exhibit the characteristics of a uniform substance (Singh and Singh, 1983). The composition dependence of the physical properties of such blends tend to follow the linear blending rule. However, incompatible polymers can undergo phase separation on blending, unless suitable compatibilizers are used. Poor interfacial adhesion between the individual phases will prevent achievement of mechanical integrity adequate for useful application (Ochigbo, 2008).

In the manufacture of rubber products, the blending of rubbers produces new materials with a wide range of applications because they have the potential to combine the attractive properties of constituents in the blend, when compared with the economical and technical uncertainties associated with synthesizing new polymeric materials. (Arayaprane, 2007).

Elastomers blends are frequently used in the rubber industry to obtain best compromise in compound physical properties, process-ability and cost. A blend can offer a set of properties that can give it the potential of entering a new application area not possible with either of the polymer comprising the blend. It has been already reported that the blending of natural rubber with other elastomers can improve its properties to a great extent. For example, blends of NR with Styrene

butadiene rubber (SBR) are noted for a combination of properties such as good abrasion resistance, while those with nitrile rubber (NBR) are noted for excellent oil resistance, those with chloroprene rubber (CR) are noted for good weather resistance, oil resistance, ozone and resistance to oxidative degradation. Several studies in the area of NR/EPDM blends are available in the literature with special reference to different blend ratio of NR: EPDM, which can improve excellent ozone resistance.

NR suffers from poor flame, weather, ozone, oil and thermal properties. Due to the strain induced crystallization behavior of NR, which can increase the modulus, resistance to deformation and stabilize the system by preventing the propagation of the defects without the use of highly reinforcing fillers and expensive coupling agents. CR is a homopolymer with many trans -1,4 configuration and it is able to crystallize on stretching so the gum vulcanizate have good tensile strength (Puspalatha and Sethu 2006).

1.2 NATURAL RUBBER (NR)

Natural rubber is a gummy whitish substance isolated from rubber tree of *Hevea brasiliensis*. It is a natural hydrocarbon which is made up of 2-methyl-1,3 butadiene (isoprene). It is known to contain one double bond per repeat unit. (Ese, 1999). The rubber tree flourishes in the tropics with annual rainfall of 2,000-4,000 mm evenly spread throughout the year, and temperatures ranging between 24-28°C.

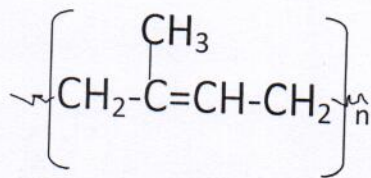


Figure 1.1; Structure of Natural Rubber (made up of 2-methyl-1,3 butadiene (Isoprene)).

The production of natural rubber (NR) is concentrated in a few tropical countries, however, as a result of intensive breeding programs, rubber tree areas can be found in locations with an annual rainfall of as little as 1,500 mm per year and a dry season of up to five months. Natural rubber is cultivated over a long gestation period of about 6-7 years and is highly labour intensive. Trees are grown in large plantations as well as being produced by small farmers in the developing countries of Asia (Thailand, Indonesia, Malaysia), Africa (Liberia, Cameroon, Nigeria), and Latin America (Brazil, Guatemala). (Robert Brentin and Phil Sarnacke, 2011).

The latex of NR is described as suspension of tiny particles of rubber in aqueous medium, containing 35% of rubber, 60 % water, resin, fatty acids, proteins and other substances. High water content can be eliminated from the latex through the use acetic acid, a process which makes the emulsion to coagulate leading to the separation of water from the solid rubber. (Ese, 1999).

A premium grade of natural rubber, pale crepe, can be obtained through different processes. The latex is treated with 0.5% sodium bisulphate after dilution to prevent enzymatic darkening and addition of chemical bleaching agents, such as Xylyl-mercaptan is added. Extremely pale rubber can be obtained by removing the colouring pigment (β -carotene) through partial coagulation, after which it is coagulated and passed through rollers, and then smoked dried at a temperature less than 40⁰C for about six days. (Ese, 1999).

1.3 NEOPRENE (POLYCHLOROPRENE)

Neoprene, the world's first fully commercial synthetic elastomer, was introduced by DuPont in 1931. Since then it has established an enviable reputation for reliable service in many

demanding applications. Neoprene, made from chloroprene monomers, may be a homo-polymer consisting of only one chloroprene units or the polymer may be polymerized to contain sulphur and/or 2, 3 co-monomers such as di-chloro 1, 3-butadiene. Neoprene is a true multipurpose elastomer with its balance of inherent properties, which include:

- Outstanding physical toughness
- Wider short- and long-term operating temperature range than general-purpose hydrocarbon elastomers
- Resistance to hydrocarbon oils and heat
- Resistance to ozone, sun and weather
- Better flame retardant/self-extinguishing characteristics than exclusively hydrocarbon-based elastomers. (Carol Spaner, 2008).

1.3.1 Composition and Uses of Polychloroprene

Most chloroprene processes are based on butadiene. Butadiene is converted into the monomer 2-chlorobutadiene-1. Chloroprene is produced from 2-chlorobuta-1, 3-diene. CR rubber is classified as general purpose, adhesive and liquid dispersion types.

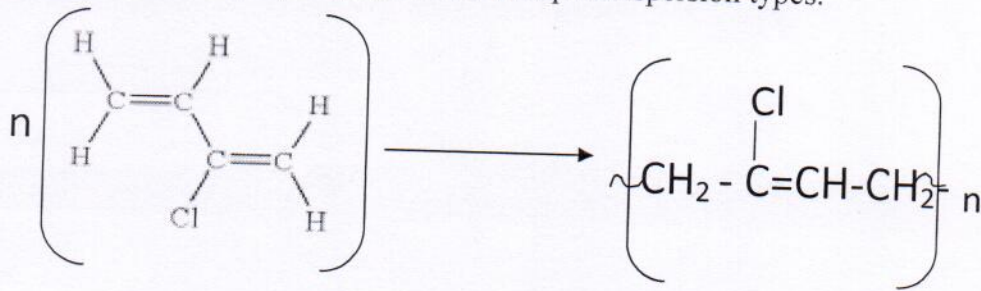


Figure 1.2: Structure 2-chlorobuta-1, 3-diene and Polychloroprene

General-purpose types are used in a variety of elastomeric applications, particularly molded and extruded goods, hoses, belts, wires and cables, heels and soles of shoes, coated fabrics, and gaskets. The adhesive type is used in solvent-based adhesives that are characterized

by quick set and high bond strength. The liquid dispersion types are generally used in adhesives, binders, coatings, dipped goods, waterborne adhesives, elasticizers, and foams. (Robert Brentin and Phil Sarnacke, 2011).

Neoprene rubber is a premium grade synthetic rubber sheet made from Chloroprene polymer. It displays good physical properties with moderate resistance to ozone, temperature and some petroleum based fluids. Neoprene rubber sheet is also available with cotton or nylon monofilament reinforcement for improved tear strength and gasket stability (Carol Spaner, 2008).

1.4 COMPARATIVE PROPERTIES OF NATURAL RUBBER AND POLYCHLOROPRENE

1.4.1 Tensile strength (stretching ability)

NR possesses excellent green strength. For an unfilled NR the tensile strength lies within the range of 15-20MN/m. the excellent strength of NR are mainly from its ability to crystallize at high strain and this is made possible as a result of it very uniform high cis-poly-isoprene molecule between neighboring molecules of chains resulting in a large increase in hysteresis and ultimate strength. (Ese, 1999).

Neoprene on the other hand is composed of 2-chloro-1, 3-buta diene, it is very tough and rigid. The structural rigidity (high trans content) of neoprene enhances quick strain-induce crystallization. (Carol Spaner, 2008).

1.4.2 Tear Strenght

Natural Rubber has very good tear resistance, especially at a very high temperature while neoprene, been has lower tear resistance. The different between NR and CR may be basically

ascribed to the tendency of natural rubber to crystallize, which helps to improve its tensile strength and also impede tearing, most importantly at point of high stress. (Ese, 1999) and (Carol Spaner, 2008).

1.4.3 Abrasion and Wear Resistance

The abrasion resistance of natural rubbers is not as good as that of neoprene except at very high temperature, though it is known that the abrasion resistance of rubbers decreases rapidly at very high temperature but in case of natural rubbers, it decreases less rapid than in the synthetic rubbers. Silica fillers or some grades of carbon black fillers can help improve the wear and abrasion resistance of rubbers. (Ese, 1999)

1.4.4 Resilience

Resilience rubbers is the rubber that have “snap” or a fast come back and generates relatively heat when flexed. This is one of the major properties of natural rubber which makes it useful in making truck tires and other heavy duty tires (Ese, 1999). Neoprene also has good fatigue resistance properties. Neoprene is mostly use where cut growth resistance is the primary requirements. In such case it must contain N772 or N774 {SRF} carbon black, or blends with N990 CMT carbon black for low modules and high elongation. Up to 20 parts precipitated silica with a dispersing aid will also assist any polymer grade with thioureas should be avoided.

A very good antioxidant / antiozonant system should be present in such compound.

1.4.5 Electrical Resistance

Neoprene, being of high polarity when compared with totally hydrocarbon based elastomer; it is not normally considered a primary insulating material (Carol Spaner, 2008).

Natural rubber on the other hand is a good electron resistance rubber. It possessed a resistivity of the 10^6 Ohm-cm (Ese, 1999).

1.4.6 Tack

NR possesses a very good tack property and does not need the addition of tacking agent during compounding. Good tacking ability is however necessary in tube to rubber or rubber to metal bonding or in the formation of composite structure so also formation of blends (Ese, 1999). Neoprene can only provide high level of building tack in the presence of plasticizers. It should also be noted that an aromatic oils are more prone to cause troublesome roll sticking than other types such as wood resin, naphthenic and esters (Carol Spaner, 2008). Natural rubber bonds excellently to most rubber and metal surfaces (Ese, 1999).

1.4.7 Oil Resistance

Natural rubber has very poor oil resistance; it is highly reactive towards petroleum oil, vegetable oil and organic solvent. Unvulcanized raw material natural rubber rapidly dissolves in organic oil and solvents, while the processed (vulcanized) NR is highly swollen by organic fluids. Neoprene on the other hand is not easily affected by oil; it has very good resistance to vegetable oil, hydrocarbon oil, and many organic solvents. This property is basically due to its low level of conjugation, inherent toughness and polarity.

1.4.8 Oxidation and Ozone Resistance

The presence of unsaturation in NR makes it highly susceptible to oxidation and ozone attack. Natural rubber in its low state (unprocessed) is readily attacked by oxygen; this could be directly or due to the presence of some oxidizing agents such as; metals and metals oxides. Poly-

chloroprene has been made optimized by the addition of antioxidants in combination with selected waxes. Therefore oxidizing agent like metal oxide has no effect. Natural rubber has extremely poor resistance to ozone attack in its unsaturated state but when saturated it does not suffer attack from ozone. Neoprene has less double bonds and it is believed that the lesser the double bonds the lesser the rubber is attacked by ozone, which is further broken down into other fragments by continuous depolymerization.

1.5 RUBBER COMPOUNDING AND COMPOUNDING INGREDIENTS

Vulcanization is the introduction of cross links in rubbers, thereby making it have considerable effect on its chemical and mechanical properties. Other additions are therefore needed to be added to improve its industrial and mechanical applications.

Therefore compounding of rubbers is the technology which involves careful selection of materials, chemical compounds and the process of incorporating them into a rubber to produce a homogeneous mixture which must be suitable for further shaping product. (Sharma, 2007)

Typically the addition of two or more polymers, materials or ingredients into a polymer is considered compounding. This process changes the personalities of a given polymer and determines the end use of the new polymer. Compounding is also described as mixing of the raw rubber with other ingredients so as to improve the property of the rubber to a desired product suitable for particular use. This is done during rubber breakdown presses.

In compounding, it is necessary to know the service condition to which the rubber product is to be exposed and the ease in manufacturing the items needed for compounding (Sharma, 2007)

The two major types of crumb rubber compounding methods are; continuous and discontinuous method. The discontinuous system is fairly old and in most cases referred to as Bandury-mixer or Roll-mill. Capacity for discontinuous system ranges from 2050 to 5,000kg/hr and a sizable investment are required, however an efficient processing system will allow the compound to operate economically at high capacity. Continuous compounding system on the other hand, has the capacity of about 2,500nkg/hr. for quality with less batch-to-batch variation than discontinuous system (Brydson, 1978).

1.5.1 Reinforcing Agents

The tensile strength of rubber and other polymer can be improved by addition of some inorganic chemicals. Chemicals like carbon black, clay Zinc Oxide, magnesium carbonate and other inorganic compounds added to rubber which helps improve the strength of the rubber such that when extended, it can withstand a pull of 4,000 pounds to a square inch. This type of reinforced polymers are extensively use in tire industry. Carbon black is the most commonly used reinforced agent, it majorly has higher influence in the strength of polymers (Sharma, 2007).

Fillers used in rubber production incused carbon black, lime stone, talk barites, zinc oxide etc. Fillers help improve the hardness and serve as diluents. Manufacture investigated the possibility of diluting rubber at a time when the cost was comparatively high. They surprisingly observed that hardness, strength and resistance to abrasion were greatly improved by adding them (Sharma, 2007).

1.5.2 Softeners

In applications like toy balloons, softeners are added to soften them so that they may be easily inflated. Softening agents in general used are liquid paraffin, paraffin wax and acid (Utpal, 2007)

They are used for a number of reasons. Some of these reasons are:

- to decrease the viscosity and thereby improve the workability of the compound
- to reduce mixing temperature and power consumption
- to reduce hardness
- to reduce low temperature brittle point
- to aid in the dispersion of fillers
- to reduce mill and calender shrinkage
- to provide lubrication and aid in extrusion and molding

1.5.3 Dispersing Agents

The particle size of solid materials added to latex must usually be made as small as possible to ensure intimate contact with the rubber particles. Solid materials are usually added to latex as dispersion. The material to be added is mixed with dispersing agents in deionized water and ground to a small particle size in a ball mill or attritor. In these devices stones or other hard pebble-sized materials are made to tumble and mix with chemicals reducing them to very small size (Utpal, 2007).

1.5.4 Mastication and Mixing

Mastication is a process of softening the rubbers, it involves the Combination actions of heat and mechanical break down rubbers of long polymer chains and it is usually applied on raw rubbers although not restricted to it alone.

The break down through mastication of rubbers brings about a permanent change in the rubbers. (Ese, 1999).

The result of mastication of rubbers includes:-

- Production in elasticity
- Production in strength property
- Render it very soluble in organic solvent
- Make it softer and cause it to flow more easily than the original state
- Render it tacky such that article can be built up from several piece or layers of masticated rubbers.

1.6 VULCANIZATION OF RUBBER BLEND

Vulcanization is a chemical transformation of rubber which involves the insertions of chemical cross link (bonds) in between a long rubber molecule. It ultimately involves the transformation of the largely plastic and highly deformable rubbers into fairly rigid three dimensional structures. However, vulcanization does not only involve the transformation, it also includes the various ways and method by which the transformation occurs. (Ese, 1999).

Vulcanizations have many effects on rubbers some of which includes:-

- Increase in tensile strength, tear strength abrasion resistance, fatigue life and toughness.
- Increase in static and dynamic modulus.
- Decease in hysteresis, permanent set and friction co-efficient.

Hysteresis is a measure of the deformation energy that is not stored or borne by the network chain instead is converted to heat

- Increase in resistance towards oxygen, ozone, and weather.

- It increases the dimensional stability and also increases its heat stability.
- It decreases the solubility toward organic/inorganic solvents.
- Improves rigidity and glass transition temperature. (Ese, 1999).

1.6.1 Vulcanizing Agents

Vulcanizing agents help increase the retractile force and reduces the amount of permanent deformation remaining after removal of the deforming force by the insertion of cross-links between polymer chains forming a cross-linked molecular network.

The crosslink may be a group of sulfur atoms in a short chain, a single sulfur atom, carbon-to-carbon bond, a polyvalent organic radical, an ionic cluster, or a polyvalent metal ion. The process is usually carried out by heating the rubber (mixed with vulcanizing agents) in a mold under pressure. (Utpal, 2007)

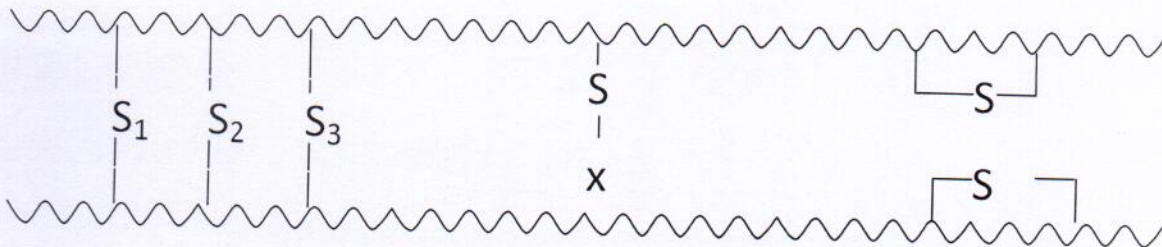


Figure 1.3; Structural features of a sulfur- vulcanized natural rubber network

1.7 MECHANICAL PROPERTIES OF POLYMER

Mechanical properties of polymer can be attributed to their behaviour under stress; these are properties that enable a polymer scientist or engineer get information about the effective use of such polymer (Coleman, 1999)

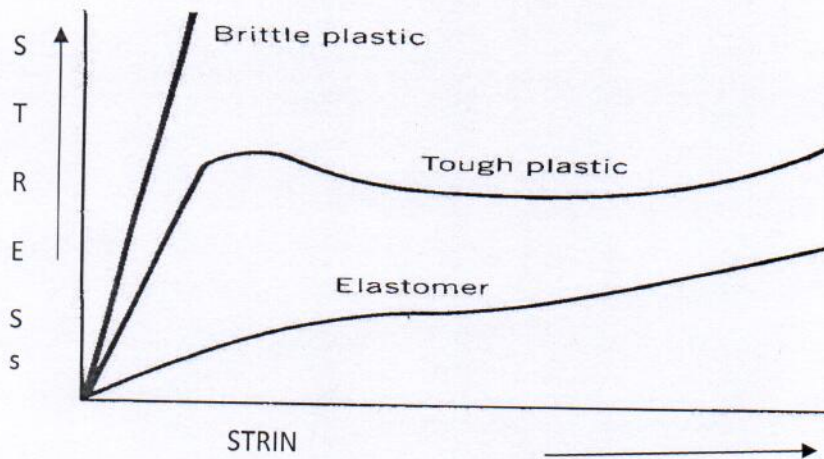


Figure 1.4; Stress- Strain behaviour of polymer

The Stress- Strain behaviour of polymer is synonymous to that of metal as shown from figure 4 above.

Curve (1) is for brittle polymer, (2) plastic, and it could be highly elastic, while Curve (3) shows rubber-like elasticity, large recoverable strain at low stress levels. Polymers that exhibit curve (3) are said to be elastomers. (Leonid, 2009)

1.7.1 Strain

Strain is the fractional deformation produced in a body when it is subjected to a set of deforming force (Gaur, 2000). It is the change in length of a body per unit amount of force.

A body subjected to force tends to undergo some changes in size, shape or both, an elastic materials tends to resist deforming force and when the deforming force is removed, there is tendency for such body to return to its original state of zero energy or stable equilibration and this tendency is due to the deformation to restore the body to its original state.

1.7.2 Stress

Stress is used to express the loading in terms of force applied to a certain cross-sectional area of an object. From the perspective of loading, stress is the applied force or system of forces that tends to deform a body.

1.7.3 Elasticity

Elasticity is the property of a body by which it tends to regain its original shape or size on the removal of deforming forces.

Under normal condition, it is not that possible to have a perfectly rigid body, all bodies tends to deform under the action of force with some highly deformed while some are less deformed. When a body has been deformed though the application of force, it is possible for such a body to regain its original shape on the removal of the deforming force (Gaud, 2000)

Natural rubber has excellent elastic properties especially when vulcanized. It shows a wide range of mechanical properties and it is one of the few elastomers which possess high strength and low hardness while Neoprene has a rigid structure as a result it has low resistance to deforming force. (Gaud, 2000).

1.7.4 Hooke's Law

It is a principle of physics that states that; the force needed to extend or compress a material by some distance is proportional to that distance. (Petroski, 1996).

Hook's law shows a single relationship between stress and strain. According to this law "Strain is proportional to the stress producing it within elastic limit"

i.e. Stress \propto Strain

$$\frac{\text{Stress}}{\text{Strain}} = E$$

Where E= Young Modulus.

1.7.5 Tensile Strength

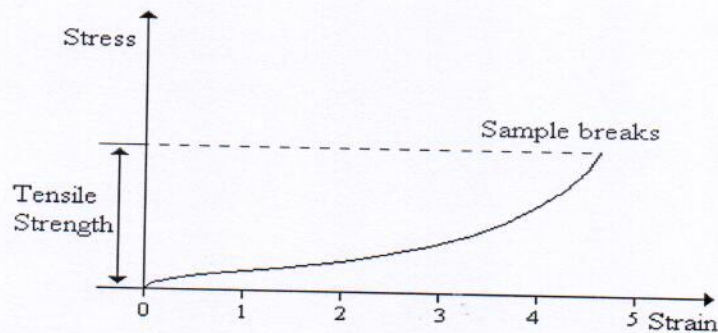


Figure 1.5; tensile strength curve of polymer

It is a strength which must be overcome for a material to break. It is expressed in Pascal or psi (pounds per square inch).

$$1\text{Mpa} = 145\text{ psi}$$

Ultimate tensile strength (UTS) often shortened to tensile strength (TS) or ultimate strength is the maximum stress that a material can withstand while being stretched or pulled before failing or breaking (Degarmo, Black & Kohser 2003). Tensile strength is distinct from compressive strength. Some materials break sharply, without plastic deformation, in what is called a brittle failure. Others, which are more ductile, including most metals, experience some plastic deformation and possibly breaking before fracture. (Smith & Hashemi, 2006).

Tensile strength is an important property of polymer in which they undergo when stretched, take for instance; fibers must have good tensile strength.

1.7.6 Elongation-at-Break.

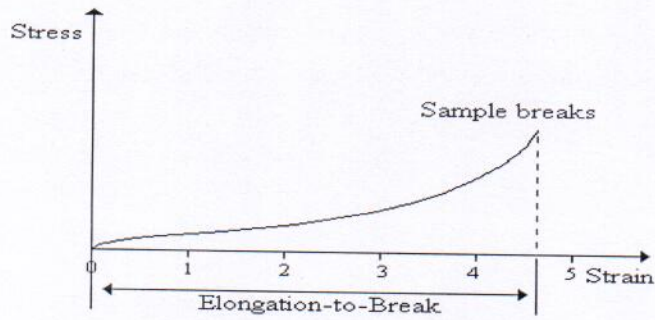


Figure 1.6; stress-elongation curve of polymers

The elongation-at-break of many materials is the strain on a sample when it breaks; it is usually expressed as a percent. Elongation-at-break is sometimes referred to as ultimate elongation. Elongation-at-break is less useful in fiber but highly relevant in elastomers. (Gilmer Williams, 1996).

1.7.7 Young Modulus

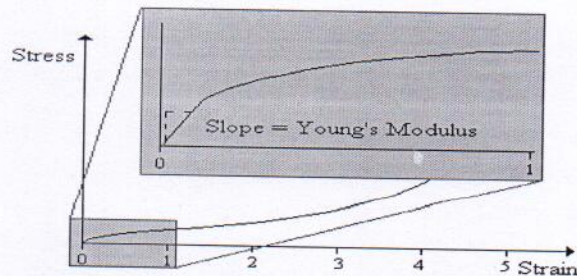


Figure 1.7; modulus curve for polymer

Young modulus is the ratio of stress to strain. It is usually called Modulus of Elasticity or sometimes called the tensile modulus.

Young modulus is the slope of a stress- strain curve. Strain- strain curves often are not strength – line plots, indicating that the modulus changing with the amount of strain. In this case the initial slope usually is used as the modulus as is illustrated in the graph in figure 1.7.

Fibers and other rigid materials like metals have high young modulus, while elastomers have low values and plastics lie in between (Ebewele, 2000)

Young's modulus is calculated by drawing a tangent to the initial linear portion of the stress-strain curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. For purposes of this calculation, the tensile stress shall be calculated by dividing the load by the average original cross section of the test specimen. (IPC-TM-650).

$$\text{Young's modulus} = \frac{\frac{\text{(load at point on tangent)}}{\text{(original width)(original thickness)}}}{\frac{\text{(elongation at point on tangent)}}{\text{(initial gage length)}}}$$

1.7.8 Thermoplastics

They are polymers that soften reversibly when heated (harden when cooled). When such polymers are heated at elevated temperature, the inter-chain bonds are weakened and this permits the deformation of the polymer at a very low stresses, but once they are cooled, the inter- chain bond is then harden, this type of property is common among linear polymers and few branched structured.

1.7.9 Thermosetting Polymer (Thermoset)

These are polymers that harden permanently when heated. Thermoplastics are known to be harder, they are more dimensionally stable and they are more brittle plastics. Examples of thermosets are; - alyresins, eposides, vulcanized rubber e.t.c.

1.8 SCOPE OF THE PRESENT WORK

The applications of NR are wide and this includes; tyres, automobile part, engineering, medicine, and pharmaceuticals. This is so because of its high tensile strength, high tear resistance, availability and minimal effects of contaminants. A large proportion is however found only in manufacture of tires. This limitation in scope of application is due to its poor resistance to oxygen, ozone, oil and hydrocarbon solvents, the high permeability to gases, bad odour and allergic complaints are added challenges.

CR rubber however, possesses some strength, oil resistance, inflammability, increased resistance to oxygen and ozone along with resistance to aging and weathering. It also finds considerable applications in areas such as; belt, wire, cable jacketing, coated fabrics, inflatable hoses and adhesives.

The relative susceptibility of NR is to agents of degradation when compared with CR may be due to the electron donating methyl group attached to the carbon bearing the double bond in its molecule as opposed to electron withdrawing chlorine atom attached to the carbon atom bearing double bond in CR.

The aim of this project is to produce new set of blends of NR and CR which may possess a compromise of physicochemical properties of the two polymers. With this, wider applications of the blends may be achieved.

The work is therefore designed to:

- To formulate scope for blending of NR and CR rubber.
- to determine the mechanical properties of Natural Rubber/Neoprene blends,

- to provide some design guidelines for the development of polymer blends,
- to determine the compatibility of the two polymers
- to determine the most compatible blend ratio which may be compared with other engineering tools in terms of strength, corrosion resistance and so on.

The overall aim of the work is to produce blends which should diversify and improve on the areas of NR and CR rubber applications due to broad property range and high performance blend production.

CHAPTER 2

2.0 APPARATUS, MATERIALS AND METHODS

2.1 Apparatus

1. Two-roll-mill (By Techno/ DIBE 4 Rue Alfred Nobel 37150 Blere Made in France.
2. Pocket Durometre Hardness Tesater ZHT2093. Made in England.
3. Oven GRIFFEN 300FC. Isuzu Seisakasho CO-LTD Tokyo Japan.
4. Zwick/Roell Z005: 300 series Electromechanical Tester. Made3 in England.
5. Metal Metrolac Jar.: Made in Nigeria.
6. Measuring Cylinder SPG100ml Graduated Made in England.
7. Metrolac-Zeal SPGRT6628 Made in Nigeria.
8. Plastic Bowl Made in Nigeria.
9. Weighing Balance: Sartorius Ag. Gottingen BP1215. Made in England.
10. Petri Dish (Glass).
11. Stop Watch: 31305 model Made in China.
12. Grinder-Apex Grinder MESH 100.
13. Micrometer Screw Guage. 196A6Z Made in Germany.
14. Michrotome (Punch): Model Dumb-bell Puncher C88036.
15. Flat bottom Glass Dish.
16. Desicator Product Number-Z553808 Made in England.
17. Conical Flask: Pyrex SPG100ml graduated Made in Eng7land.
18. Hot plate Clifton hot plate HP1.30E Made in China.
19. Compression moulding machine: TECHNON LOIRE PLCN50T-3P

20. Rheometer: ALPHA TECHNOLOGYES. Oscillating Disc Rheometer ODR 200
MODEL Made in France.

2.2 Materials

1. Water
2. Ammonia
3. Natural Rubber Latex
4. High Aromatic hydrocarbon
5. Xylyl Mercaptan
6. Stearic acid
7. Magnesium Oxide
8. Zinc oxide
9. Sulphur
10. MBTS & CBS
11. Octylated diphenylamine
12. Mixed diacyl-p-phenylene diamine
13. Carbon Black
14. Process Oil
15. Crumb rubber processed from CAR 10Grade
16. MBTS
17. CBS
18. TMQ
19. Octylated diphenylamine
20. Diacyl

21. Some selected Waxes

22. Magnesia.

2.3 GENERAL FORMULATION

The formulation used for the two rubbers are presented on table 2.2 below. All measurements were carried out in part per hundred of rubber (Phr).

Table 2.1 Formulation for NR and CR

NR Formulation	Phr	CR Formulation	Phr
NR	100	CR	100
Carbon Black	30	Carbon Black	30
Processing Oil	5	Processing Oil	5
Zinc Oxide	5	Zinc Oxide	5
Acid	2	Acid	2
TMQ	1	TMQ	1
MBTS	2	MBTS	2
CBS	1	CBS	1
Sulphur	1.5	Sulphur	1.5
Magnesia	-	Magnesia	4

For purpose of obtaining 100% NR/0%CR and 0%NR/100%CR all compounding ingredients with the curing agents were mixed and milled using two roll mill. For the compounding of the

blends master batch of NR and CR formulation were used in exclusion of the curing agents; MBTS, CBS and sulphur.

2.4 BLEND RATIO

The blends were prepared using the proportion presented on Table 3.3

Table 2.2; Blend ratios

BLEND	NR%	CR%
A	100	0
B	80	20
C	60	40
D	40	60
E	20	80
F	0	100

2.5 COMPOUNDING PROCESS

The mixing, compounding and vulcanization, of the polymers were done using the two-roll-mill by following the steps below:

PROCEDURES:

- 1 All materials were weighed, the two-roll-mill was prepared and allowed to work until its temperature rose to about 150°C.

2. The weighed Rubber was masticated using the two-roll-mill. This helps exposed the surface area of the polymer to aid easy incorporation of the compounding ingredients.
3. After proper mastication the Compounding ingredients were added in the following order Processing oil, Stearic acid, Zinc oxide, Carbon black and TMQ.
4. Further mastication carried out allows adequate incorporation of the ingredients until homogenous product was achieved.
5. The material obtained was then allowed to cool down.

Table 2.3; compounding ingredients used

MATERIAL	AMOUNT (Phr)
Natural Rubber	100
Carbon Black	30
Processing Oil	5
Zinc Oxide	5
Stearic Oxide	2
TMQ (2,2,4-trimethyl-1,2dihydroquinoline)	1

Note: Phr; means part per hundred.

2.6 VULCANIZATION PROCESS

The vulcanization process of all the blends were carried out by using the master batches previously produced and this was done by adding MBTS, CBS and Sulphur to them. The formulation used for the blends is presented on Table2.4 using the steps below:-

1. Compounded Natural Rubber and Polychloroprene were adequately weighed other vulcanizing ingredients too were weighed.

2. The weighed Natural rubber and Neoprene were masticated in the two-roll-mill simultaneously.
3. Then vulcanizing ingredients were added according to the following order MBTS, CBS, Sulphur and Magnesia
4. The resulting compound was properly masticated till homogenous compound was obtained.
5. Other blends were also prepared by using the same procedure.
6. Curing of the compound was done one after the other in the compression molding machine at 150⁰c.
7. After the curing, the compression molding machine was switched off and the mould was removed.
8. Then it was prepared for testing for Hardness, tensile strength testing and elongation determinations.

Table2.4; formulation used for Natural Rubber/Polychloroprene Vulcanization

COMPOUNDS	AMOUNT (Phr)
Rubber Blends	100
MBTS (2,2'-Benzothiazyl di-sulfide)	2
CBS (N-Cyclohexyl-2-benzothiazole sulfenamide)	1
Sulphur	1.5
Magnesia	4

2.7 TESTING AND DETERMINATION OF PARAMETERS

The mechanical analysis of the blends was carried out using **Zwick/Roll (Z005 model)** tensile testing machine which experimentally determine the stress-strain behavior of the blends. The determination was done by cutting 3mm thickness, 10 mm cross-sectional area, 30mm of length and 4mm specimen width using a bumb-bell mechanical cutter. The test piece was clamped in the already prepared tensometer (which has been prepared for experimental determination). The test piece was then extended by the machine and the following mechanical properties were recorded on the chart given in the appendix.

- Stress-%elongation curve
- Maximum force (load) applied on the specimen
- Tensile strength of the specimen
- Elongation at maximum
- Elongation at break
- Tear force
- Tear strength were directly read from the system

The same procedure was used for all test pieces from the resulting blends.

2.8 HARDNESS

All values of hardness in IRHD were derived using Durometer (Digital Hardness Tester) GE'S Dyna POCKET is a small integrated hardness tester which operates according to the dynamic rebound method (ASTM A956 and DIN EN ISO 18265).

The compact design allows easy on-site hardness testing on solids, non-transportable components and even on position difficult to access by other hardness tester. This was used to determine the hardness of the resulting blends.

CHAPTER 3

3.0 RESULTS AND DISCUSSION

3.1 Latex and Crumb Characterization

The natural rubber used in this analysis is of "CAR 10 GRADE" produced at the Rubber Research Institute of Nigeria. The properties of these materials are presented on Table 3.1. Quality assurance parameters such as; total solids content (TSC) and dry rubber content (DRC) were found to conform with technically specified rubber used in rubber technology. (Egharevba et al, 2011).

Table 3.1; Latex and crumb characteristics.

PARAMETERS	RESULTS
Total solid content (TSC) (%)	39.66
Dry rubber content (DRC) (%)	32.6
Plasticity retention index (PRI Min)	50
Original plasticity (P _o) (mm) (Crumb)	45
Plasticity Retention index (PRT) (%) (Crumb)	92.2
Dirt content (%) (Crumb)	0.17
Volatile matter (%) (Crumb)	0.81
Ash content (%) (Crumb)	0.36
Colour	Brown

The dry rubber content of 32.6% is excellent and can be used for work like this. The crumb rubber for this work has Plasticity Retention Index (PRI) of 92.2% and low dirt content of 0.17% which were found to be excellent for our blending process (SAR, 1998).

Table 3.2 shows the comprehensive results of mechanical relationship of the blends (B, C, D and E) and that of A (100%NR) and F (100%CR).

Table 3.2 Mechanical properties of the Blends

BLENDS	% CR	Tensile strength (Mpa)	Modulus(100) (Mpa)	Modulus(200) (Mpa)	Modulus(300) (Mpa)	Tear Force (N)	Tear Strength (MPa)	Elongation At Break (%)	Hardness IRHD
A	0	17.4 ±2.2	1.0 ±0.0	2.0 ±0.0	3.2 ±0.1	139.1 ± 17.2	17.4 ±2.2	869.9 ±41.8	53
B	20	23.3 ± 1.5	1.0 ±0.1	1.7 ±0.2	2.6 ±0.2	186.0 ±11.8	23.3 ±1.5	1291.7 ±88.0	50
C	40	8.6 ± 0.4	1.7 ±0.3	3.2 ±0.1	4.6 ± 0.0	128.0 ±6.0	8.6 ±0.4	600 ± 32.7	58
D	60	15.3 ±4.8	2.9 ±0.6	5.6 ±0.7	8.6 ±0.5	122.5 ±38.4	15.3 ±4.8	503.7 ±144.0	63
E	80	9.2 ± 2.5	3.6 ±0.4	6.6 ±0.7	9.0 ±0.0	92.2 ±24.9	9.2 ±2.5	295.8 ± 96.1	70
F	100	9.6 ± 1.3	4.2 ±0.9	7.3 ±0.2	10.3 ±0.0	115.6 ±16.1	9.6 ±1.3	252.4 ± 72.2	85

3.2 Tensile Properties

The Ultimate Tensile Strength (UTS) or the Tensile Strength being the strength that must be overcome before material breaks were determined for 100%NR/0%CR, 0%NR/100%CR and the blends. Results obtained are presented on Table3.3 and further analysis is shown on Figure3.3.

Table3.3; Tensile properties of the blends

BLEND RATIO	TENSILE STRENGTH(Mpa)
NR /CR	
A 100/ 0	17.4 ±2.2
B 80/ 20	23.3 ± 1.5
C 60/ 40	8.6 ± 0.4
D 40/ 60	15.3 ± 4.8
E 20/ 80	9.2 ± 2.5
F 0/ 100	9.6 ± 1.3

From Figure 3.1, it can be seen that the tensile properties of the blends decrease with increase in % chloroprene although not in a regular pattern and blend B which is the 80% NR/20%CR was surprisingly found to have highest tensile strength of all the blends produced. From this observation we may conclude that, the stretching ability of natural rubber was enhanced with 20-30% CR blend. Such a blend can also offer low cost commercial polymer in terms of tensile strength when considering the cost of CR. The least Tensile Strength was observed for sample E (20% NR/80%CR) while sample D gave a value close to that of 100% NR. Higher amount of NR gives better resilience tensile strength and even compression set.

(Pupalatha and Sethu, 2006).

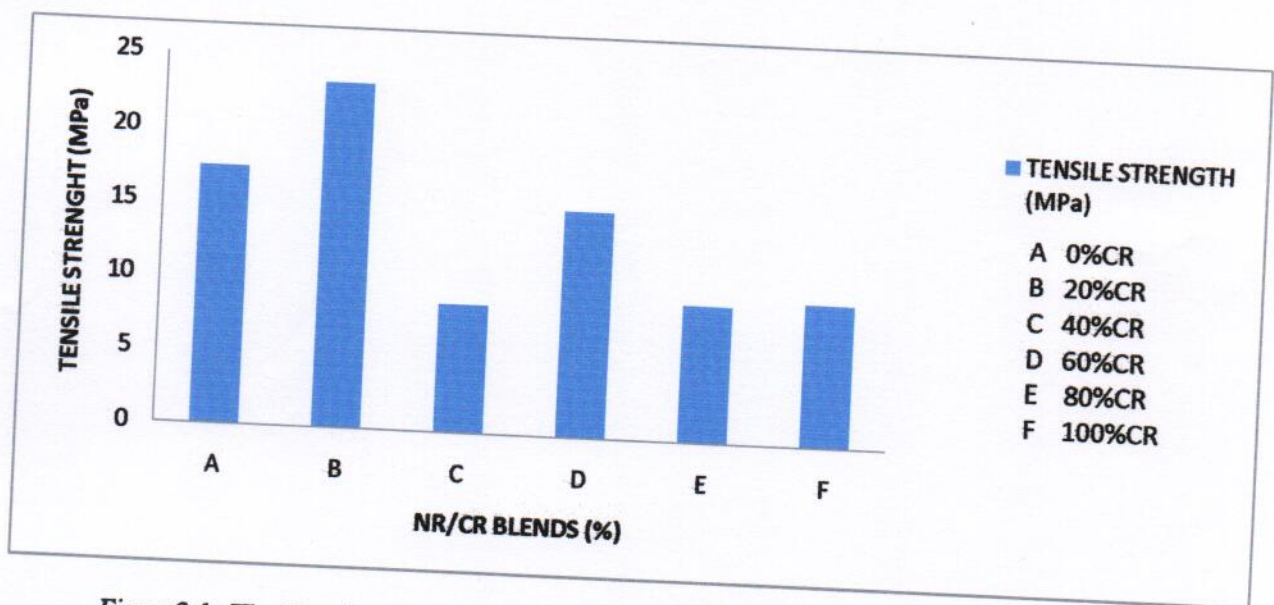


Figure 3.1; The Tensile Strength of blends at different % CR composition

3.3 Percentage Elongation

The result shows that 100%NR gave a value of 869.9 ± 41.8 while 0%NR/100%CR gave 252.4 ± 72.2 being the lowest value recorded. The decrease in elongation at break with increasing %CR was noticed although a deviation was observed for sample B being 1291.7 ± 88.0 . For any

blend the Stress-Strain behaviour shows a linear relationship as shown in Appendix A. The strain starts from 40% elongation and increases linearly to about 290% until the material breaks at 869.9% strain. The blend is said to have a curve for high rubber – like elasticity with a large coverable strain at low stress level (leonid 2009).

Table 3.4 Elongation at Break.

BLEND RATIO	ELONGATION AT BREAK (%)
NR /CR	
A 100 /0	869.9 ±41.8
B 80/20	1291.7 ±88.0
C 60/40	600 ± 32.7
D 40/60	503.7 ±44.0
E 20/80	295.8 ± 96.1
F 0/100	252.4 ± 72.2

Blend **B** also show the same kind of rubber – like elasticity behaviour, the strain (% elongation) increase linearly from 25% elongation showing a straight line to about 400% elongation and eventually breaks at 1291.7% elongation. This is said to be highly elastic and this type of curve can only be obtained for elastomers. (Leonid, 2009). Blend **C** tends to deviate partially from the rubber- like behaviour. The initial strain for its straight line starts around 50% elongation which shows a linear behaviour to about 350% strain and it reaches its break point at about 600% elongation. This could also be said to be elastic but relatively low when compared with blends **A** and **B**. Blend **D** shows a plastic-like elasticity. It increases linearly from 225% elongation to 425% elongation. It reaches its break point at 503.7% elongation which shows that it has some elastic properties but tougher and with higher resilience when compared with blend **A**, **B**, and **C**

(Billmeyer, 1984). Blend E as obtained from appendix E shows a brittle – plastic – like behaviour, it is very hard, tough and shows little elastic properties (Billmeyer, 1984). Its linear curve extends between 28.8% strain to 220.5% strain and reaches its break point at 295.8% elongation. Blend F which is NR/CR100 from Appendix F linearly increased with increase in strain from 50% strain up to 275% strain until it breaks at 252.4% elongation which shows that the material is hard and brittle.

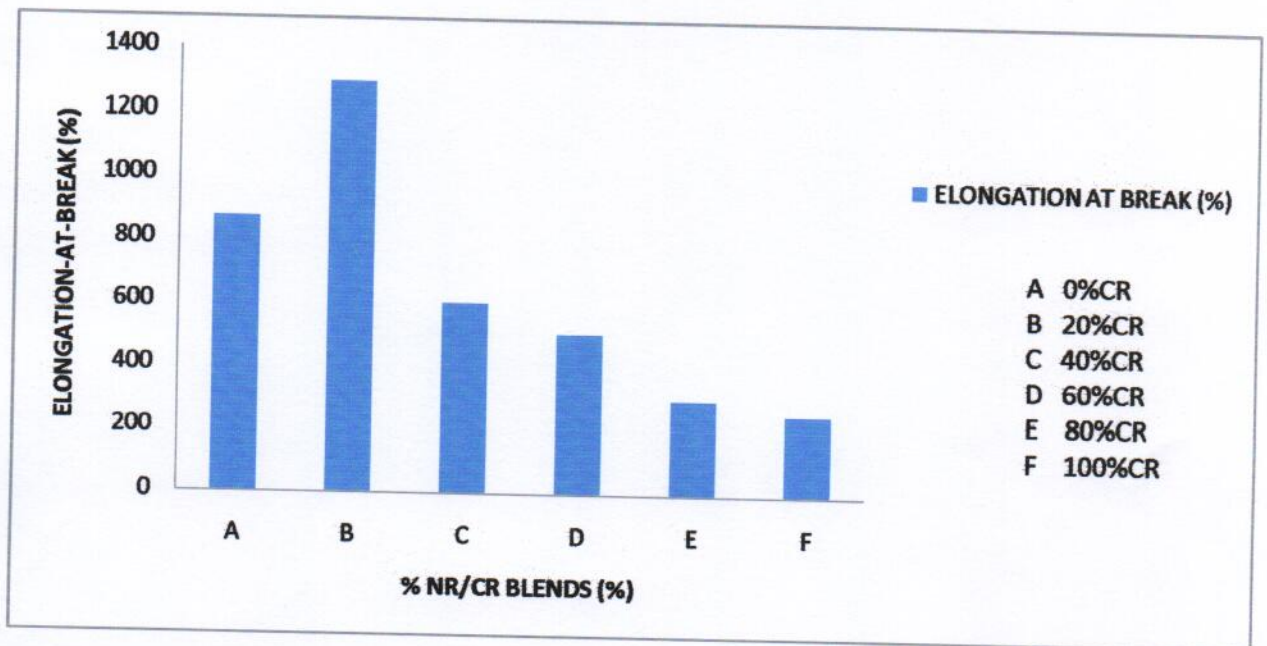


Figure3.2; Elongation at break of blends at different % CR composition

It can be seen that initially the polymers shows high % elongation but increase in the % CR results into a plastic – like behaviour and latter result in a brittle materials (Leonid, 2009).

3.4 Hardness

The value of the hardness of 100%NR, 100% CR and that of the blends are presented on Table3.4 below. The result shows that 0%NR/100%CR gave the highest value of hardness with the least being 50 IRHD recorded for 80%NR/20%CR (Malomo, 2015).

Table 3.5; Hardness value for NR/CR blends.

BLEND RATIO	HARDNESS IRHD
NR /CR	
A 100/0	53
B 80/20	50
C 60/40	58
D 40/60	63
E 20/80	70
F 0/100	85

From the data it can be deduced that, the hardness of the polymer blends increase with increase in polychloroprene rubber. Although it was initially found to be negligible indicating that low amount of polychloroprene will offer lower cross-linkages in the natural rubber while increasing amount of polychloroprene increases the cross-linking density of the material, consequently the higher the resistance to indentation and abrasion resistance (Zorge, 2011).

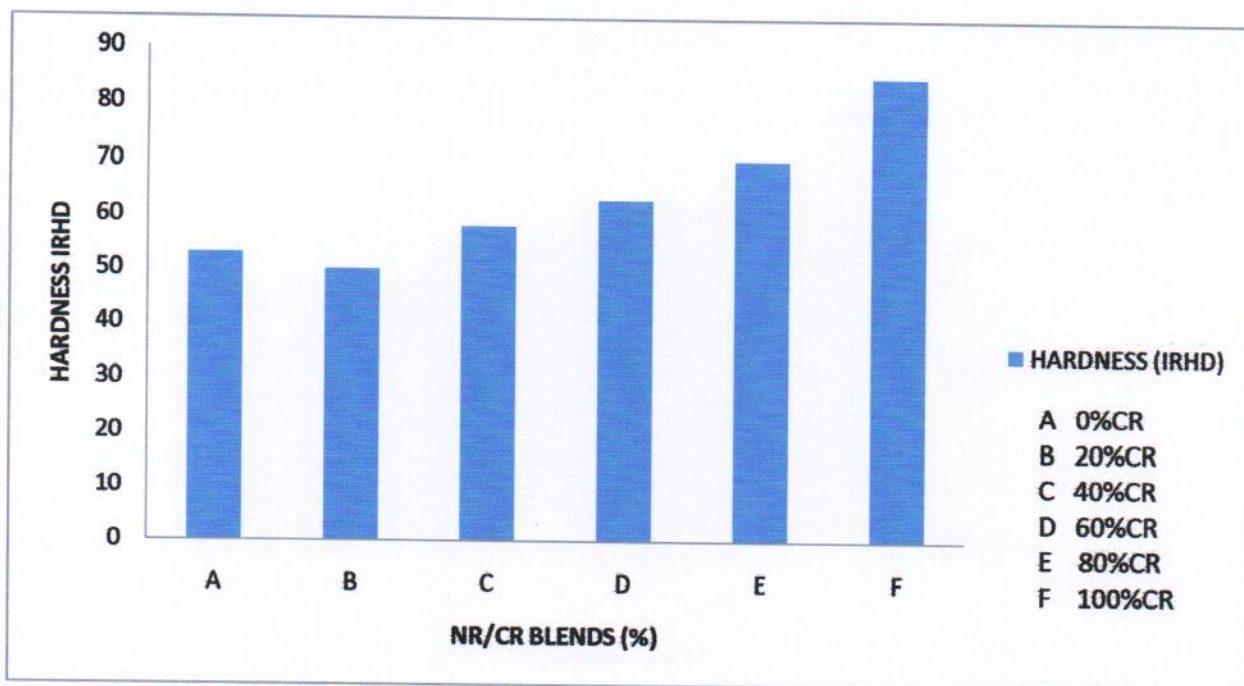


Figure3.3; Hardness of various of blends at different % CR composition

3.5 Modulus

A graph of modulus at 100,200 and 300 % elongation for different %CR and the blends is shown on figure3.4. The results show that at any %CR composition, the modulus increases with %elongation. It was also found that the modulus increases with increase in % CR composition as presented on table3.6. The increased self crystallizing property of NR at greater elongation (Strain) may be responsible for this observation (Malomo, 2015).

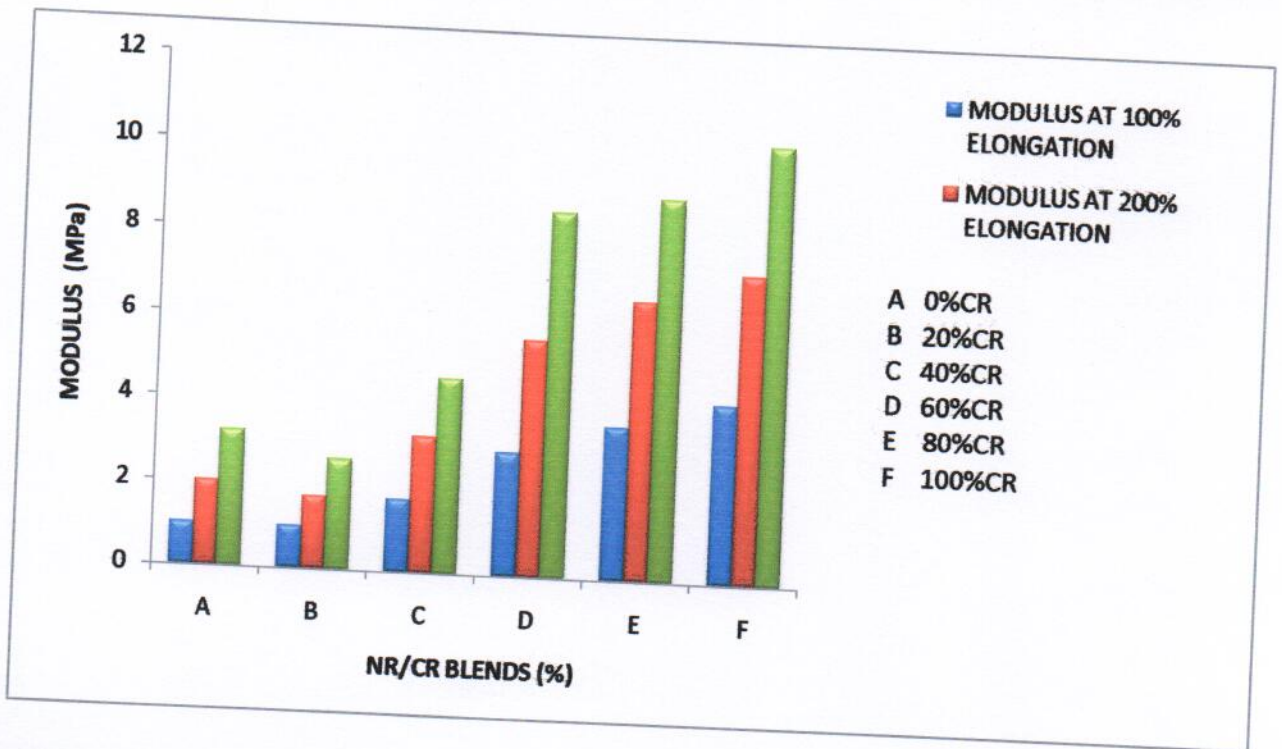


Figure 3.4; Modulus of various blends at different % CR composition.

The plotted values of modulus is given in table 3.6 below.

Table 3.6: Modulus vales at 100, 200 and 300 percent elongation

BLENDS	% CR	Modulus(100) (MPa)	Modulus(200) (MPa)	Modulus(300) (MPa)
A	0	1.0 ± 0.0	2.0 ± 0.0	3.2 ± 0.1
B	20	1.0 ± 0.1	1.7 ± 0.2	2.6 ± 0.2
C	40	1.7 ± 0.3	3.2 ± 0.1	4.6 ± 0.0
D	60	2.9 ± 0.6	5.6 ± 0.7	8.6 ± 0.5
E	80	3.6 ± 0.4	6.6 ± 0.7	9.0 ± 0.0
F	100	4.2 ± 0.9	7.3 ± 0.2	10.3 ± 0.0

CHAPTER 4

4.1 CONCLUSION AND RECOMMENDATION

This work shows that adding CR to NR produces blends in with a drop in the tensile property of NR was observed. Natural Rubber vulcanizates are known to have high tensile strength over a wide range of hardness. The high strength of NR may be due to crystallization of the polymer chains at high strain (Chukwu, 2002). From the result of this work, it is evident that NR and CR mixed homogenously during the blending process. The various blends produced should be suitable for wider NR applications such as product required for greater resistance to hydrocarbon oil, solvents and weathering.

Test report

Customer : MR RAJI ABIMBOLA .O
 Job no. : NR100/CR0
 Test standard : DIN 53504,05/1994
 Material : RUBBER
 Specimen type : DUMBELL
 Pre-treatment : NATURAL RUBBER/POLYCHLOPRENE RUBBER BLENDS
 Tester : F C IBEH (Polymer Technologist). contact: +234-805-1575101
 Notes... : Rubber Research Institute of Nigeria, Iyanomo
 Test speed : 200 mm/min

Test results:

Nr	Date	Clock time	l_0 mm	F_{Max} N	σ_{max} MPa	ϵ_{Fmax} %	F_R N	σ_R MPa	ϵ_R %	σ_{50} MPa	σ_{100} MPa	σ_{200} MPa	σ_{300} MPa
1	4/14/2015	8:51:32 AM	30	153.1	19.1	901.1	153.1	19.1	901	0.5	1.0	2.1	3.2
2	4/14/2015	8:55:00 AM	30	144.3	18.0	886.1	144.3	18.0	886	0.5	0.9	2.0	3.3
3	4/14/2015	8:58:13 AM	30	119.9	15.0	822.4	119.9	15.0	822	0.6	1.0	2.0	3.2

Nr	σ_{500} MPa	L_c mm	L_0 mm	a mm	b mm	A_0 mm ²
1	6.4	30.00	30.0	2	4	8.00
2	6.5	30.00	30.0	2	4	8.00
3	6.3	30.00	30.0	2	4	8.00

Series graph:

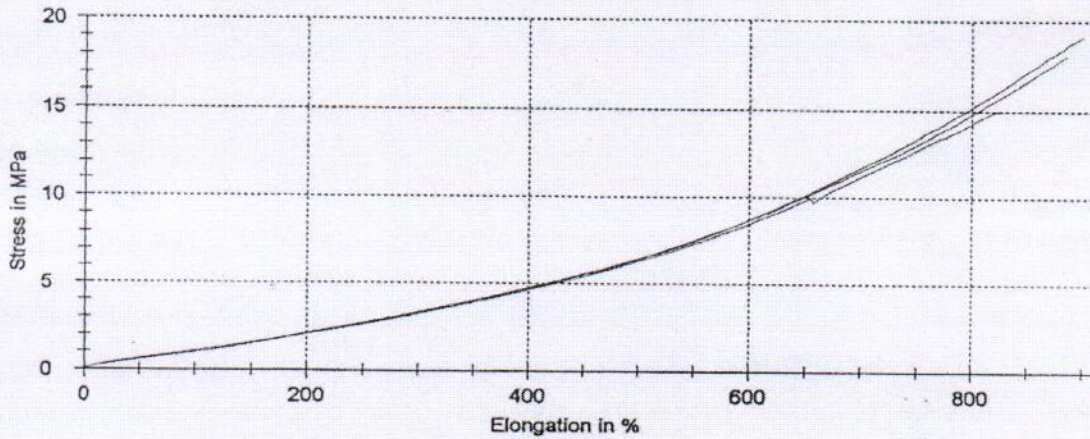


FIGURE A; Test Report for sample A

Test report

Customer : MR RAJI ABIMBOLA O
 Job no. : NR80/CR20
 Test standard : DIN 53504,05/1994
 Material : RUBBER
 Specimen type : DUMBELL
 Pre-treatment : NATURAL RUBBER/POLYCHLOPRENE RUBBER BLENDS
 Tester : F C IBEH (Polymer Technologist), contact: +234-805-1575101
 Notes... : Rubber Research Institute of Nigeria, Iyanomo

Test speed : 200 mm/min

Test results:

Nr	Date	Clock time	l_0 mm	F_{Max} N	σ_{max} MPa	ϵ_{Fmax} %	F_R N	σ_R MPa	ϵ_R %	σ_{50} MPa	σ_{100} MPa	σ_{200} MPa	σ_{300} MPa
1	4/14/2015	8:17:21 AM	30	172.5	21.6	1194.5	172.5	21.6	1195	0.5	0.9	1.7	2.6
2	4/14/2015	8:21:11 AM	30	193.8	24.2	1366.1	193.8	24.2	1366	0.6	1.1	1.9	2.8
3	4/14/2015	8:25:31 AM	30	191.8	24.0	1314.5	191.8	24.0	1315	0.5	0.9	1.6	2.4

Nr	σ_{500} MPa	L_c mm	L_0 mm	a mm	b mm	A_0 mm ²
1	4.9	30.00	30.0	2	4	8.00
2	4.9	30.00	30.0	2	4	8.00
3	4.5	30.00	30.0	2	4	8.00

Series graph:

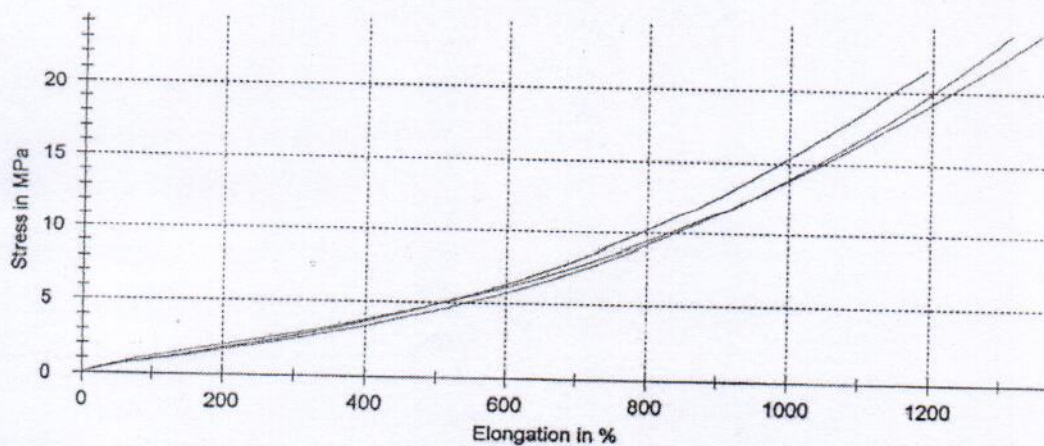


FIGURE B; Test Report for sample B

Test report

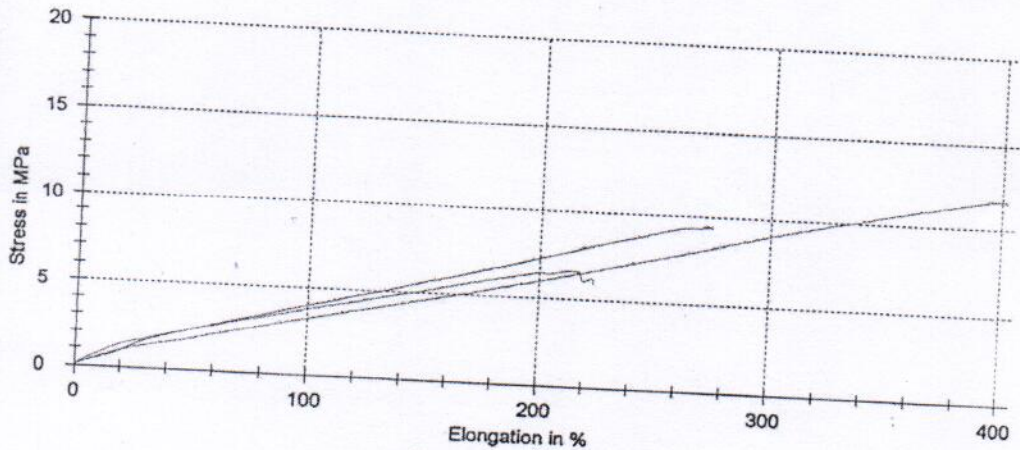
Customer : MR RAJI ABIMBOLA,O
 Job no. : NR20/CR80
 Test standard : DIN 53504,05/1994
 Material : RUBBER
 Specimen type : DUMBELL
 Pre-treatment : NATURAL RUBBER/POLYCHLOPRENE RUBBER BLENDS
 Tester : F C IBEH (Polymer Technologist). contact: +234-805-1575101
 Notes... : Rubber Research Institute of Nigeria, Iyanomo
 Test speed : 200 mm/min

Test results:

Nr	Date	Clock time	l_s mm	F_{Max} N	σ_{max} MPa	ϵ_{Fmax} %	F_R N	σ_R MPa	ϵ_R %	σ_{50} MPa	σ_{100} MPa	σ_{200} MPa	σ_{300} MPa
1	4/14/2015	8:31:32 AM	30	94.0	9.4	273.5	94.0	9.4	273	2.3	3.9	7.3	-
2	4/14/2015	8:33:32 AM	30	116.2	11.6	401.1	116.2	11.6	401	1.8	3.2	6.0	9.0
3	4/14/2015	8:36:32 AM	30	66.4	6.6	212.8	66.4	6.6	213	2.3	3.7	6.5	-

Nr	σ_{500} MPa	L_c mm	L_0 mm	a mm	b mm	A_0 mm ²
1	-	30.00	30.0	2.5	4	10.00
2	-	30.00	30.0	2.5	4	10.00
3	-	30.00	30.0	2.5	4	10.00

Series graph:



abimbola 3.zs2

FIGURE E; Test Report for sample E

Test report

Customer : MR RAJI ABIMBOLA.O
 Job no. : NR0/CR100
 Test standard : DIN 53504,05/1994
 Material : RUBBER
 Specimen type : DUMBELL
 Pre-treatment : NATURAL RUBBER/POLYCHLOPRENE RUBBER BLENDS
 Tester : F C IBEH (Polymer Technologist). contact: +234-805-1575101
 Notes... : Rubber Research Institute of Nigeria, Iyanomo
 Test speed : 200 mm/min

Test results:

Nr	Date	Clock time	l_0 mm	F_{Max} N	σ_{max} MPa	ϵ_{Fmax} %	F_R N	σ_R MPa	ϵ_R %	σ_{50} MPa	σ_{100} MPa	σ_{200} MPa	σ_{300} MPa
1	4/14/2015	8:43:28 AM	30	123.3	10.3	298.9	123.3	10.3	299	1.8	3.4	7.1	10.3
2	4/14/2015	8:47:06 AM	30	97.1	8.1	169.2	97.1	8.1	169	3.1	5.2	-	-
3	4/14/2015	8:48:17 AM	30	126.4	10.5	289.1	126.4	10.5	289	2.4	3.8	7.4	-

Nr	σ_{500} MPa	L_c mm	L_0 mm	a mm	b mm	A_0 mm ²
1	-	30.00	30.0	3	4	12.00
2	-	30.00	30.0	3	4	12.00
3	-	30.00	30.0	3	4	12.00

Series graph:

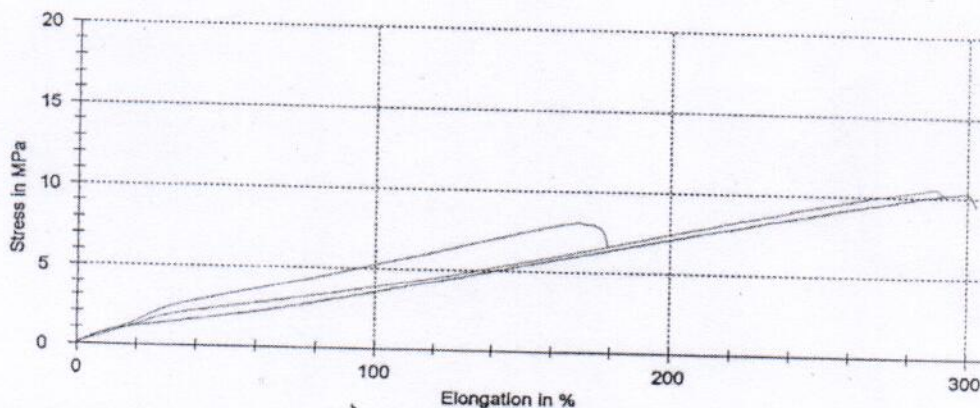


FIGURE F; Test Report for sample F

ABBEV	UNIT	NAME
Specimen identifier		specimen iden
Date		date
Clock time		clock time
L_s		parallel specimen length
F_{max}	N	maximum force
F_{max}	MPa	tensile strength
E_{max}	%	elongation at maximum
FR	N	tear force
SR	Mpa	tear strength
ER	%	elongation at break
S_{50}	Mpa	force at travel preselection
S_{100}	Mpa	force at travel preselection
S_{200}	Mpa	force at travel preselection
S_{300}	Mpa	force at travel preselection
L_c	mm	crosshead guage length
L_o	mm	gauge length
A	mm	specimen thickness
B	mm	specimen width
A_o	mm ²	cross-section

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