FEDERAL UNIVERSITY OF AGRICULTURE
ABEOKUTA NIGERIA

69TH INAUGURAL LECTURE

NATURAL RUBBER: ADDING VALUE FOR LIFE SUSTENANCE AND ECONOMIC DEVELOPMENT

by

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The Vice-Chancellor

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Protocol

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Members of my family,
All Invited Friends and Guests,
Ladies and Gentlemen,
Gentlemen of the Press and
Great Funabites

Preamble

It is with utmost gratitude to the Almighty Allah that I stand before this cosmopolitan audience to deliver my inaugural Lecture, which is the 69th in Federal University of Agriculture, Abeokuta (FUNAAB) and the 4th from the College of Physical Sciences. I consider it a privilege, indeed, that this inaugural lecture titled- Natural Rubber: Adding Value for Life Sustenance and Economic Development is being delivered by me.

Let me at the onset thank you all for leaving your tight schedules to be part of this event. For the benefit of our guest from outside the

University, I wish to briefly explain that inaugural lecture is an opportunity for the University community outside one's department, friends, family and the public to know more about the research and teaching activities that one has engaged in over the years. Traditionally, all Professors are expected to give inaugural lecture on any aspect of their work as their contributions to knowledge. With the opportunity I'm having today, I will try my best to present my lecture in a very simple way that will be understood by the audience here gathered.

1.0 Prologue to the lecture

1.1 My Entry into the World of Chemistry – the University of Lagos days

Like many in the Nigerian academia in their various fields, my entry into the world of Chemistry was a default event, in the sense that I did not apply to study Chemistry in the university, ab-initio. By God's providence, I got admitted into the University of Lagos (Unilag) in1987 to acquire university education,where I graduated with a B.Sc.Ed. (Hons) Chemistry Education in 1991. Having entered in that fray, I had the privilege of excellent mentors:Profs Jide Alo, Oluwole Familoni, Duro Ajeyalemi (late) and others who, with their thoroughness gave me grooming and tutelage in General Chemistry Education and scientific methods, which prepared me for a future that has manifested into breakthrough now. So my forays into Polymer Chemistry and the ripples towards Chemical industry sustainability have turned out to be an expression of what had been deeply rooted in my subconsciousness from an early stage in academic life.

1.2 The Benin Era: Acquiring Polymer Chemistry Skills and New Vistas

I got to the University of Benin (Uniben) in 1992 to study for a Master's degree in Industrial Chemistry, on the assistance of my uncle, Professor Ghalib Ayoakin Akinlabi, who lectures at the Optometry Department in Uniben. He introduced me to the then Head of Chemistry Department, Prof. Felix. E. Okieimen, who immediately signed me in as one of his Masters students, and that was the genesis of what I am today. When I related this development to one of my friends, he laughed at me and kicked against my choice and he asked me 'What are you going to do with Chemistry?'- This became a challenge since that time until you hear my conclusion. One course in Uniben Chemistry that aroused my curiosity and indeed caught my fancy and fascination was 'Rubber Chemistry and Technology'- taught by Dr. J. D. Ajayi (late). As I got more and more exposed to the nitty-gritty of the course, my curiosity finally found passion. Indeed, my first taste of independent scientific investigations was during this period and I have remained grateful to my Project Supervisor, Prof. Felix Ebodaghe Okieimen, who as at today has produced 39 PhD graduates in which 14 of us are Professors, while some are Executive Directors and Heads of different establishments. Looking back at that period, I realized that my interest in Polymer Chemistry was subconsciously seeded, albeit in an ill-defined manner at that stage.

Rubber Research Institute of Nigeria- Benin City

My love for Polymer Science and Rubber Chemistry, courtesy of Prof. Felix Ebodaghe Okieimen took me to the Rubber Research Institute of Nigeria (RRIN) in Benin City, after completing my Masters programme. There, I met the then Acting Director, Dr Ayoola B. Fasina (late), who was also a Polymer scientist and a friend Dr. Stephen Uzochukwu (aka Stinpex). He saw my interest in rubber and decided to have me as a 'Casual Staff' in 1996, since there was Government embargo on appointment then. I started as a Research Officer 7 and within two years I had turned around the Rubber Technology Laboratory and End Use unit of RRIN into an enviable busy section full of research activities which became a center point for all visitors to RRIN. This made the Acting Director to nominate me for a Rubber Technology course in France and another Masters in Rubber Science, Computing and Engineering in the United Kingdom (UK), which were sponsored by the Professional Association of Natural Rubber in Africa (ANRA) under the funding of United Nations Office for Project Services (UNOPS). All these trainings actually solidified my interest and prepared me to achieve my break throughs in the field of Polymer Chemistry.

1.3 Research at FUNAAB

After returning to RRIN from the University of North London, (UNL), UK, I continued with my PhD research works making use of modern equipment in UNL and the ones donated by UNOPS to RRIN.During my Ph.D. oral defense in Uniben, Prof. I. C. Eromosele came as External Examiner. He was highly impressed with my work and found my trainings over the years a good fit for the vision/mission of FUNAAB and decided to invite me to FUNAAB, where I am today as a Professor of Industrial and Applied Chemistry. This is the brief background to my research focus and the ancillary ripples that surround issues of Polymer Chemistry. For this inaugural lecture, I will focus on these areas:

- My contributions to the Chemistry and Polymer world. My work in Polymers covers polystyrene, polypropylene, polyethylene, funtumia elastics and gum Arabic but I decided to concentrate on Natural Rubber (NR) due to its versatile usability and availability here in Nigeria and in Ogun State in particular. It was grown at Ikene Remo by late Obafemi Awolowo.)
- Entrepreneurship of my research findings
- Advice to researchers and government

2.0 **Definition of terms**

2.1 Growth in Chemistry

- In recent years people have started showing interests in new areas in Chemistry like: Green Chemistry, Nanochemistry, Nuclear Chemistry, Biomolecular Chemistry, Space Chemistry, Polymer Chemistry, Computational Chemistry,etc. The link within all these areas of chemistry is their application into real life situation to solve problems, creating a new idea and generally improving the world's living conditions.

Industrial chemistry

- is where chemistry is being applied for a particular purpose. It is the conversion of theoretical and laboratory studies to human daily use to improve living standards. It is a very wide aspect but will like to throw more light on the POLYMER aspect.
- Polymers can either be natural or synthetic. Natural ones are God's gift like proteins, starch, Deoxyribonucleic acid (DNA), Natural Rubber while the synthetic ones are man-made and may be sub divided into thermoplastics, thermosets and elastomers. Classification of Polymers is as shown in Figure 1 .

Examples of Polymers:

- Natural polymers include- Proteins, DNA, starch, cellulose, Ribonucleic acid (RNA), natural rubber, etc
- Syntheticpolymers- Poly(styrene), poly(propylene), poly(vinyl chloride), poly(acrylonitrile), etc

Sustainability-

- The process of maintaining change in a balanced fashion, in which the exploitation of resources, orientation of technological development and growth are maintained. It is the ability to maintain at a certain rate or level andthe ability to continue at the same level for a period of time:

Economic development-

- Growths of industries are often associated with new or pioneer industries that did not exist in the past. The *growth* in capacity to supply a new commodity, a mechanism in which profitability plays a central role as source of capital and as a stimulus to investment. Growth is expected to link *industrial* activity with primary sector activity, domestic and foreign trade with long service life activity. Growth is expected to lead to an increase in per capita income of individual and of a country.

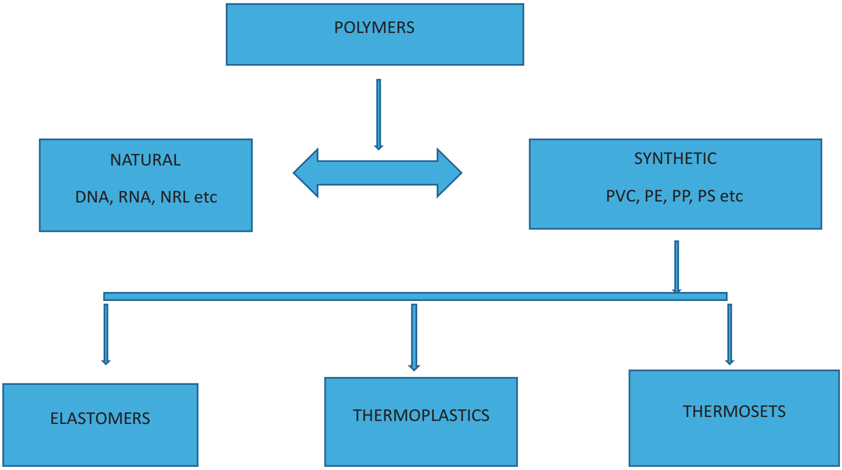


Fig. 1: Showing classification of polymers

An elastomer is a polymer with viscoelasticity (i.e., both viscosity and elasticity) and with weak intermolecular forces, generally low Young's modulus and high failure strain compared with other materials. Elastomer is a polymer that displays rubber-like elasticity. They are polymeric materials that can be stretched up to 10 times their original size with little or no permanent deformation (will return to almost its original position when the stress is removed). Example is rubber band, bicycle and car tubes etc.

Thermoplastics are plastic material which melt into a soft, pliable form above a certain temperature and solidify upon cooling. Thermoplastics can be re-melted and re-shaped for a number of times. They are usually stored in the form of pellets prior to the molding process. Common examples of thermoplastics includesacrylic, polyester, polypropylene, polystyrene, nylon, teflonetc for producing plastic bottles, 'pure water', plastic bags, plastic buckets etc. These materials see a wide variety of use in manufacturing products from clothing and non-stick cookware to carpets and laboratory equipment.

Thermosets are alternately known as thermosetting plastics or thermosetting polymers are materials which remain in a permanent solid state after being cured. Polymers within the material cross-link during the curing process to form an unbreakable, irreversible bond. This means that thermosets will not melt even when exposed to extremely high temperatures. They are polymeric materials that can not be remelted once set and degrade on application of heat. Common examples of thermoset plastics and polymers include epoxy, silicone, polyurethane and phenolic. In addition, some materials such as polyester can occur in both thermoplastic and thermoset versions

3.0 Research Activities

Below are some of the researches I have conducted on polymers particularly on natural rubber while some researches are still on-going. I have grouped them into major areas as follows:

(i) Eliminating some inherent problems associated with natural rubber- coagulation, preservation, concentration, etc

(ii) Chemically modifying natural rubber to improve and increase its usability- chlorination, Nitration, amination, alkylation, arylation, epoxidation, halogenation, depolymerization, deprotenization, etc

(iii) Solving problems of low resilience high molecular weight natural rubber by producing Liquid Natural Rubber (LNR), Low Molecular Weight Natural Rubber (LMWNR), Epoxidized Natural Rubber (ENR), Depolymerized Natural Rubber (DNR), Epoxidized Depolymerized Natural Rubber (EDNR), Deprotenized Natural Rubber (DENR), Epoxidized Deprotenized Natural Rubber (EDENR), etc

(iv) Making natural rubber a multifunctional material-:blending natural rubber with some synthetic polymers, treatment of water, effluent, oil spillage with natural rubber and photocatalytic degradation of dyes with modified rubbers.

(v) Using natural rubber in drug delivery systems- The trial of rats with antidermal, anti fungi. Insulin for diabetes and Baobab extracts.

(vi) Local sourcing of additives for natural rubber compounding- Utilization of clays, cement kiln dusts, agricultural waste materials/biomas asbiodegradable compounding rubber additives and gradual replacements of rubber additives with local agricultural waste materials

(vii) Characterization of oils from rubber seeds and their potential uses. Preparation of alkyd resin, binders, perfumes, mosquito repelling creams, pomades processing oils, etc

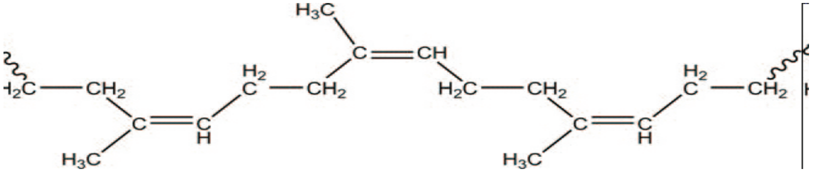
(i) Eliminating some inherent problems associated with natural rubber latex

The production of latex is a characteristic feature of many plants, but latex containing rubber in large quantities occurs only in the species of families Moraceae (mulberry), Euphorbiaceae (*Hevea brasiliensis*), Apocynaceae (*Funtumia elastica*) and Compositae (*euthamia*kyhosia)(Vambeilen and poirier, 1998). Nowadays most natural rubber latex is derived from the species *Hevea*

Brasiliensis of the family Euphorbiaceae (Akinlabi *et al* 1997).

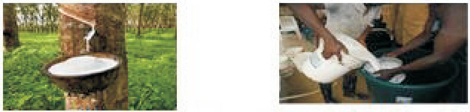
(a) Natural Rubber Latex

Natural rubber can be described as a material which is highly elastic and can be stretched to ten times its original length without breaking and will return quickly to its original length on releasing the stretching force. Natural rubber is poly-cis-1,4-isoprene and is highly stereo regular.



Chemical structure of cis -1,4-polyisoprene(Akinlabi,2001)

Freshly tapped Hevea latex (Fig.2) contains not only rubber particles but also non-rubber particles dispersed in an aqueous serum. The ratio of rubber to non-rubber components varies with source. The rubber component from Hevea rubber tree is an entirely more than 98% of cis-1,4-polyisoprene which is unable to crystallize under normal conditions, hence, it exists as an amorphous, rubbery material.



Collection of Latex Natural rubber latex

Cup Lumps / Coagula Crumb rubber

Figure 2 showing pictures of latex, cup lumps. Coagula and crumb rubber

Natural rubber is used extensively in many applications and products, either alone or in combination with other materials. In most of its useful forms it has a large stretch ratio, high resilience, and is extremely waterproof (Akinlabi *et al,* 2001). The rubber latex is a colloidal dispersion of solid particles of the polymer *polyisoprene* $(C_5H_8)_n$ and its content in the emulsion is about 34%. Natural rubber possesses properties that are entirely different from other engineering materials. It has excellent strength, which attributed to its ability to crystallize at high strain. It also possesses excellent resistance to tear, particularly at elevated temperature. Furthermore, the abrasion and wear resistance of natural rubber are fairly good. Natural rubber possesses a unique combination of low hysteresis and elasticity therefore, high resilience at low strain. It also possesses extremely good electrical resistance. Natural rubber has a very good tack resistance however it has poor resistance to oil and organic solvents. It is susceptible to oxidation and ozone attack due to the presence of unsaturation (Akinlabi *et al*, 2001). Interestingly, elasticity is one of the fundamentally important properties of NR due to the freely rotating links of its long chain molecules (Okieimen and Akinlabi 2002). Rubber is unique in theextent to which it can be distorted, and the rapidity to which it recovers to its original shape and dimensions. It is, however, not perfectly elastic and the rapid recovery is not complete. Part of the distortion is recovered more slowly and another part is retained. The extent of this permanent distortion is known as permanent set which depend on the rate and duration of applied force. The lower the force, and the longer it is maintained, the greater is the permanent set (Okieimen *et al* 1991. Akinlabi *et al* 1997, Akinlabi *et al* 2000 Aigbodion *et al* 2001 and Okieimen and Akinlabi 2002).

Natural rubber latex (NRL) has pH of 6.5-7.0 with density of 0.98 g/cm^3 and typically contains: Rubber- 34% (by weight), Proteins- 2-3%, Resins- 1.5-3.5%, ashes- 0.5-1%, Sugar-1.0-2.0%, Sterol glycosides- 0.1-0.5%, water- 55- 60%.

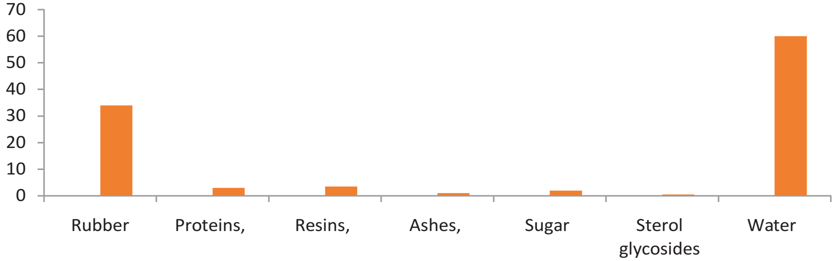


Fig. 3: Showing composition of natural rubber latex (Akinlabi *et al* 1997).

This fresh latex is not always utilized in its original form due to its high water content and susceptibility to bacterial attack, hence there are needs to solve these problems. One of the ways to solve these problems is to reduce the water content of the rubber. This has been done using concentration methods by adopting the following processes: Creaming, Centrifuging, Evaporation and Decantation

(b) Natural Rubber Latex (NRL) Concentration methods

The following methods were used and found to have reduced the water in the NRL:

Creaming

Fresh natural latex can be transformed to concentrated natural latex (around 60% rubber and 40% water) in order to realize economical transportation and easier latex product's preparation. The concentrated natural latex is an extremely valuable material and can be used for production of many products. Industrially, the concentrated natural latex can be prepared by centrifugation which requires an enormously expensive machine. From the ecofriendly products point of view, we thought of developing a green rubber product via creaming process. If natural rubber latex is well shaken with three or four times its volume of water and then allowed to stand, a separation into two layers usually takes place which thus effects purification of the dispersion. This phenomenon brought about creaming of NRL. During our creaming trials, we used so many additives like caustic alkali and gelatin of similar hydrophilic colloids, such as gum tragacanth, gum arabic, peptone, albumen, sodium alginate etc. However, the two promising creaming processes with a novel creaming agent were with the use of sodium alginate and hydroxyl ethyl cellulose with ammonium laurate soap. These methods gave us a good results with total solid content of 63.5% and dry rubber content of 61.4%. The properties of the creamed latex produced were well compared with concentrated latex produced from other sophisticated methods. With this, we were able to design a simple, cheap and green method that does not require expensive machine but uses water-based chemical as sodium alginate to prepare the cream concentrated latex.

Centrifuging

The natural rubber latex was processed into high quality latex concentrate of 60% dry rubber content through centrifugation. High-speed centrifugation of natural rubber latex was done at 10,000 rpm for 45 min at 4 ˚C. After centrifuging, we separated the preserved field latex into two fractions, one containing the concentrated latex of more than 60% dry rubber and the other containing the serum. Further processing for improving the centrifugation efficiency of NR latex to more than 90% and reducing the Dry Rubber Content (DRC) of the skim to less than 2% was also achieved via combination of creaming and centrifuging. The process involves effecting the clustering of latex particles in the natural rubber latex which is to be separated by centrifugation. In particular the clustering of the latex particles was effected prior to centrifuging the latex. The clustering of the latex particles was achieved by adding a latex creaming agent such as alginates, tamarind seed powder, and polyvinyl alcohol, preferably at the concentration ranges of 0.3% to 0.7% of DRC. Higher concentrations may be used without any particular advantage. This process yielded concentrated latex of up to 92%. We found that it was necessary to both preserve and concentrate the latex, so that the natural rubber latex becomes stable and contains 60% or more of rubber, thereby reducing the water content. NRL concentrates were differentiated by the methods used for concentration and the type of preservative used. High concentration was achieved by centrifugation, which is the most viable method recommended (Akinlabi *et al* 2001).

Evaporation

One of the methods employed in the production of concentrated latex has been confined to the controlled evaporation of water from the latex by the application of heat. This method is based on electrodialysis between vertical semipermeable membranes. The separation gives superimposed layers containing different concentrations. The more concentrated layers which is the rubber, being at the uppermost. This stratification is probably the result of the combined influence of the electrical potential and gravitational force under suitably chosen conditions. The colloidal particles migrate to one of the membranes under the influence of the electric field, with consequent concentration at the membrane surface. If the field strength is not too great, the particles become more under the influence of gravitational force than of the electrical force, tending to cause deposition on the membrane. The concentrated NRL tends, therefore, to move upwards along the surface of the membrane in accordance with its relative density with respect to that of the surrounding water. If this action is allowed to continue, the dispersions of different concentrations spread out through the cell liquid in the form of horizontal layers which may then be decanted.

Decantation

This is a process of removing the water from the actual rubber present when different layers have been formed. Decantation is mostly used in conjunction with centrifuging. Once two clearly distinct layers of rubber and water have seen, the separation by decantation proceeds. The rubber lays floats on the water, which makes it easier to separate (Figure 4).

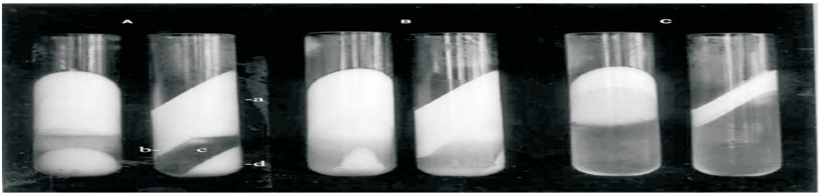


Fig. 4: Showing centrifuged latex to be decanted

© Preservation of Natural Rubber Latex

Natural rubber latex (NRL) being a stable dispersion of cis-1,4-polyisoprene has an aqueous phase and upon centrifugation it can be divided into three principal phases, namely rubber particles, aqueous phase and lutoid phase. The aqueous non-rubber phase is made up of carbohydrates, proteins and amino acids and other serum constituents as well as a range of enzymes. Non-rubber components in NR latex encourage the proliferation of bacteria that causes latex destabilisation due to their metabolic activities. Presence of bacteria in NR latex triggers a fermentation process which hydrolyses the non-rubber substances. As a result of acidic by-products (volatile fatty acids), the pH of latex decreases and neutralisation of the charges occurs, contributing to the destabilisation of NR latex particles, which thereafter gives the obnoxious odour and release acid that coagulates the latex. Hence, the need to preserve the latex. Preservation of latex can be divided into two types. Short term preservation retains field NR latex in a liquid state for a few hours or days before processing. Long-term latex preservation is a preservation system that permits the shipment and storage of liquid latex until it is ready to be processed by the product manufacturer.

Ideally, a good preservation system for NR latex should have the following criteria: (i) free of N-nitrosamines (ii) should not affect the rubber particles (iii) shouldbe water soluble (iv) should be able to preserve field latex for dry rubber production for up to two weeks and latex concentrates for at least one year with satisfactory properties (v) should not be toxic (vi) to be locally available and (vii) cost effective. In the preservation of natural rubber latex (NRL), surfactant assists in enhancing the NRL stability by maintaining the electrostatic repulsion on the surface of the rubber particles and therefore prevents flocculation of rubber molecules. Currently, chemical-based surfactants are widely being used. Surfactant is referred to as a surface active substance due to its ability to modify the surface properties between two immiscible liquids. The fact that NRL coagulates spontaneously within few hours of tapping gave us another task of how to retain its liquid form. Several chemicals and methods were investigated by using them either singly or in combinations (sodium sulfite, formaldehyde, boric acid, hydroxylamine, etc), until we were able to come up with the use of Ammonia, whichhas the best preservation for freshly tapped NRL. Ammonia(0.7%) was found to be the best among other percentages. This percentage was also found to preserve the latex from the bacterial attack (Akinlabi *et.al.* 2001). Concentrated rubber latex is a highly specified material and is characterized by a number of properties that are significant to the user. They can be stored for longer period with most of the properties remaining largely unchanged apart from properties such as potassium hydroxide (KOH) number, Volatile Fatty Acids (VFA) number and latex Mechanical Stability Time (MST) that are time dependent (Akinlabi *et. al.* 2001).

(d) Coagulation of Natural Rubber Latex

Coagulation of natural rubber latex involves the removal of ammonia, which can either be done by adding an acid or at least one salt selected from the group consisting of calcium salts and ammonium salts of nitric acid, sulfuric acid, carbonic acid, phosphoric acid, hydrochloric acid and formic acid and/or by a polymeric coagulating agent being added to a natural rubber latex under agitation to form a porous coagulated rubber. The coagulation of NR latex suspensions to produce solid NR is a well-recognized industrial practice. A range of coagulants such as acetic acid, formic acid, surfactants, and a mix of acids and salts are often used to change the ionic strength of the aqueous media. Removal of ammonia or dialysis narrows the range of pH while the addition of saline substances, *e. g.*, potassium sulfate or ammonium acetate, broadens it. These results accord with the view that the rubber particles carry an adsorbed protein layer. The delayed coagulating effect of sodium fluosilicate used in the manufacture of rubber articles, arises not merely from the gradual development of acidity, but also from the concurrent formation of zinc ions. If the zinc ions become effective at very high a pH value, the coagula may be undesirably weak. This can be prevented by the additional presence of a sparingly soluble zinc salt like zinc sulfite. The coagulanting activity of sodium fluosilicate can also be controlled by the addition of salts which decrease its solubility or its rate of hydrolysis. At ordinary temperatures ammonium persulfate is too slow to be a satisfactory delayed coagulant, but its action can be accelerated by the addition of suitable reducing substances.

Vacuum technology was also applied to coagulate natural rubber latex. Under vacuum, natural rubber particles in natural rubber latex expand, burst and coagulate. This vacuum coagulation of natural rubber latex could be controlled physically. The skim latex obtained as a byproduct during this procedure was cleaner than that obtained from other procedures; hence, this procedure would therefore reduce pollution from latex processing. The mechanism of the coagulation of latex and the theories are numerous but I have been able to conclude that the use of formic and acetic acids still remain the best and recommended due to the fact that the nonionizable component of latex, that is the hydrocarbon that constitutes the core of the latex globule will still remain intact. We eventually came up with the use of 3% formic acid (HCOOH)and5% acetic acid as the best for latex coagulation. Auto coagulation also occurs but difficult to control and takes a longertime than when acids are used. (Akinlabi *et al* 2001, Egbon *et al* 2003 and Okieimen *et al* 2003a)**.**

(e) Processing of Rubber Latex to Crumb Rubber

The latex is usually collected from the tree about 3 to 4 hours after tapping. This is followed by treatment to prevent premature coagulation and brought to a factory or a smallholder processing centre. Ammonia (about 0.7%) is usually added, being the most common stabilizer. At times, otherstabilizers such as sodium sulfite and formaldehyde are also still used. In the initial collection (3 to 4 h), about 80-85% of the latex produced by the tree is collected as latex (field latex).The latex continues to exude very slowly for several hours after the initial collection. This latex is left to coagulate spontaneously in the cup to form “field coagulum” or “cuplump”. A small amount of latex also coagulates as a thin sheet on the tapping cut to form “tree lace.” These are collected on the next tapping day and constitute about 15-20% of total yield. On arrival at the factory, the latex is sieved and blended. Field latex is either concentrated by removing part of the water to give “latex concentrate,” or it is deliberately coagulated and processed into solid dry rubber. All cuplumps are processed into dry rubber.

Concentration of the latex is usually by centrifugation but occasionally, creaming or evaporation might be used. The centrifuged latex is shipped as latex concentrate containing about 60% dry-rubber content. The remainder of the latex and field coagulum are processed into conventional types of rubber such as Ribbed Smoke Sheets (RSS), Pale Crepes (PC), and Brown Crepes (BC), or into the newer forms of Technically Specified Rubbers (TSR).

(f) Compounding of Natural Rubber

Rubber is always compounded with additives. It is through compounding that rubbers are designed to satisfy a given application in terms of properties, cost, and processability. Products manufactured from natural rubber latex tend to be very pure and have the enhanced physical properties that natural rubber latex are known for e.goutstanding elongation, tear properties, recovery etc. The colour of freshly coagulated rubber is brownish to white. It is elastic and elasticity greatly increases after vulcanization. However, it becomes a hard solid, called ebonite, when vulcanized with about 30-40 percent sulphur. Vulcanized rubber isinsoluble in most solvents and swells up only in ether, petrol and turpentine. The vulcanized rubber is highly elastic and is not very sensitive to change of temperature, whereas natural rubber is very sensitive to temperature.

Rubber is an electrical insulator and has high tensile strength. Some important physical properties which are of great importance in categorizing the grades of rubber are tear resistance, tensile strength, resilience, hardness, resistance to abrasion, reactivity with oxygen and ozone, resistance to oil and solvent and elasticity. Elasticity is a combination of (a) ability to stretch rapidly reaching high elongation, (b) high degree of stiffness when fully stretched, and (c) ability to return to its original length rapidly, when the stress is removed. Additives can include fillers which act either to enhance the rubber's mechanical properties (reinforcing fillers) or to extend the rubber to reduce cost (non-reinforcing fillers. Other additves are classified as

- Vulcanizing agents
- Accelerator:
- Activators:

- Retardants:
- Anti-degradant
- Processing aids
- Anti oxidants
- Plasticizers
- Tackifiers
- Softeners,
- thickeners,
- blowing agents, etc

(g) Characterization of latex from NIG 800 clones
There are 14 newly developed rubber clones planted in the Rubber Research Institute of Nigeria (RRIN) in early 80s and they are termed NIG 800 series. Akinlabi *et. al.*1997characterized all the 14 clones to know the quality of latex from each clone. The clones were developed in the 80s but due to seven years gestation period of rubber, tapping of the latex did not start until late 90s. We also assessed the level of Mistotles on some infested trees and quality of latex from such infested trees were characterized. For the mistletoes We could not ascertain how long they have been on the trees but we looked at their sizes and the number on each tree and group them as 1-3 (Low), 4-6 (medium), 7-9 (high) and above 9-(badly invested). Our finding showed that the number of infestation affect the quantity and quality of latex. This was done both during rainy and dry season. However, the quantity of latex was always higher in the rainy season but got washed away sometimes by rain. (Akinlabi *et. al.* 2005)

(ii) Chemically modifying natural rubber to improve on some of its limitations

The rubber industry is usually dominated by one product, tyres. *Tyr*es are used in large numbers on automobiles, trucks, aircraft and bicycles. Production of rubber goods consists of two basic steps: (1) Production of the rubber itself from the latex and (2) Processing into finished goods, which consist of (a) Compounding; (b) Mixing; (c) Shaping and (d) Vulcanizing. My research work has shown how rubber can be used in other valued products.

Limitations of natural rubber like poor resistance to oxygen, ozone, solvents, etc were improved upon through structure modifications so that there will be few site available for easy oxidation or external penetration. This was achieved through various chemical modifications via oxidation, sulphonation, nitration, epoxidation, hydroxylation, acetylation, arylation and hydrogenation.

(a) Hydrogenation- This is an addition reaction, where hydrogen was added into the rubber chain. The Rubber was hydrogenated at high temperature and pressure in presence of platinum black as catalyst to give a hard rubber used as ebonite. It is blackish brown firm and can be used for a long period..

(b) Halogenation- This is the addition of halogen (group vii elements). Rubber was reacted with halogens to form halogenated rubbers and specifically with chlorine and bromine to produce chlorinated and brominated rubbers which was found to be inelastic and amorphous in nature.

Bromination-This is more selective than chlorination because the reaction is less exothermic. Most common bromination is conducted by the addition of Br_2 to alkenesand aromatic substrates, giving rise to a host of organobromine compounds. The usual catalyst is the bromoperoxidase which utilizes bromide in combination with oxygen as an oxidant. An example of bromination can be found in the organic synthesis of the anesthetic halothane from trichloroethylene.

Organobromine compounds are the most common organohalides in nature. Their formation is catalyzed by bromoperoxidase and are estimated to release 1–2 million tons of bromoform and 56,000 tons of bromomethane annually.

(c) Hydrochlorination-Rubber was reacted with hydrochloric acid to form rubber hydrochloride, $(C_5H_8.HCl)_n$.

(d) Oxidation-When natural rubber was exposed to air, it slowly oxidizes. Oxidizing agents such as nitric acid, peroxybenzoic acid, peroxides and permanganate when used, caused oxidation of rubber and the oxidation process was promoted by copper and manganese. Normal oxidation of rubber gave unstable peroxide of rubber which was transformed into a stable oxide. Oxidation of NR was reduced and prevented by the reduction of the double bonds in the structure. This was found to reduce the number of sites available for oxygen attack.

(e) Ozonolysis- The ozonolysis of rubber ozonide confirms the fact that head and tail linking up of large number of isoprene molecules takes place in rubber. Our team obtained a glassy explosive mass of the composition $(C_5H_8O_3)_3$ by passing oxygen containing 6% ozone into a solution of rubber in chloroform. When the ozonide was decomposed by water, laevualdehyde and its peroxide, laevulic acid, succinic acid, formic acid and CO_2 were formed. These products of ozonolysis suggested that rubber was composed of isoprene units joined head to tail. The X-ray diffractive studies showed that rubber is composed of long chains of isoprene units arranged in cis-form. The chains became more parallel on stretching and were scatterly arranged. When the

rubber was cooled to 180 C, it became crystalline and non-elastic and when heated with about 1% of an organic sulphonyl chloride or an organic sulphonic acid at 120- 130 C, it was converted into a tough, thermoplastic resin, which resembles gutta-percha.

(f) Alkylation- Alkylation is the transfer of an alkyl group from one molecule to another. The alkyl group may be transferred as an alkyl carbonation, a free radical. A carbon ion or carbine (or their equivalents) alkylating agents are widely used in Chemistry because the alkyl group is probably the most common group encountered in organic molecules. Many biological target molecules or their synthetic precursors are composed of an alkyl chain with specific functional groups in a specific order. Selective alkylation or adding parts to the chain with the desired functional groups, is used, especially if there is no commonly available biological precursor. Alkylation with only one carbon is termed methylation. Alkylation of NRL was found to increase the hydrocarbon content without changing the elastic behavior of the rubber.

(g) Acetylation-Acetylation (or in IUPAC nomenclatureethanoylation) describes a reaction that introduces an acetylfunctional group into a chemical compound. (Deacetylation is the removal of the acetyl group.) Acetylation refers to the process of introducing an acetyl group (resulting in an acetoxy group) into a compound, namely the substitution of an acetyl group for an active hydrogen atom. A reaction involving the replacement of the hydrogen atom of a hydroxyl group with an acetyl group (CH_3 CO) yields a specific ester, the acetate. Acetic anhydride is commonly used as an acetylating agent reacting with free hydroxyl groups. For example, it is used in the synthesis of aspirin and heroin. Acetylation of NRL was investigated and we were able to come up with a flexible elastic material with more sites for crosslinking due to additional carbon that was added couple with the oxygen available for addition reaction.

(h) Epoxidation- Epoxidation is the addition of oxirane rings on the double bonds. In a typical experiment, 100ml of latex was measured into the beaker, then 10ml of distilled water was added, after which 10 ml of hydrogen peroxide (H_2O_2) was added to it and was placed on magnetic stirrer to make the solution even. The rubber latex was then coagulated with 10 ml of formic acid and thoroughly washed with the addition of water and pressed till the natural rubber latex was no longer acidic to litmus. Thereafter, it was cut into pieces by the use of a knife, after which it was oven dried for 3 hours at 100^0C. The same procedure was repeated for 5ml, 15ml, 20ml, 25ml and 30 ml of hydrogen peroxide (H_2O_2) and also for 5ml formic acid.

(i) Hydroxylation- Hydroxylation is a chemical process that introduces a hydroxyl group (OH) into an organic compound. In biochemistry, hydroxylation reactions are often facilitated by enzymes called hydroxylases. Hydroxylation is the first step in the oxidative degradation of organic compounds in air. It is extremely important in detoxification since hydroxylation converts lipophillic compounds into water soluble (hydrophilic) products that are more readily excreted. Some drugs (e.g. steroids) are activated by hydroxylation. The chemical concept of hydroxylation processes involves conversion of a 'CH' group into a 'COH' group. Hydroxylation is an oxidative process. The oxygen that is inserted into C-H bond is usually derived from atmospheric oxygen. Since oxygen itself is a slow hydroxylating agent, catalyst are required to accelerate the rate of the process. Hydroxylation of NRL was successfully carried out resulting in an elastic material with fewer number of double bonds than the started material. Part of the double bonds have been used in bonding with –OH-

(j) Sulphonation:- The natural rubber latex (NRL) was sulphonated using *in situ* method. The temperature of the medium was about 30 °C. In the reaction, 100ml of stabilized latex was

weighed into the beaker and placed on a magnetic stirrer to make the solution even. Fuming sulphuric acid and sulphur trioxide were added and stirred. Fuming sulphuric acid, is also referred to as *oleum*, which is a concentrated solution of dissolved sulphur trioxide in sulphuric acid. The sulphur in sulphur trioxide is electrophilic because the oxygen pull electrons away from it since oxygen is very electronegative. The double bonds in the NRL attract the sulphur and subsequent proton transfers occur to produce sulphonated rubber. The reaction was left for various hours and different concentrations were used. At the end of the reaction, the reaction was terminated by coagulating the rubber with formic acid and washed with distilled water severally till acid-free (no longer acidic to litmus) before drying at 70°C.

Findings

From the field latex collected, the characteristics of the latex are as shown in Table 1. The dry rubber content (DRC) of natural rubber latex is the actual amount of the rubber hydrocarbon present in the latex. It is the percentage by mass of coagulated latex within a specified conditions of colloidal destabilization determined according to ISO number 126:1995(E). The real rubber hydrocarbon present was 32.66%.The total solid content of natural rubber latex is the amount of solid materials present in the rubber latex which indicates that there is presence of non-hydrocarbon materials in the latex. It is very useful in determination of the non-rubber solid content present in the latex, which is 38.48%.

Table 1 Characterization of natural rubber latex used for chemical modifications

Test	**Results**
Dry Rubber Content (DRC)	32.66%
Total Solid Content (TSC)	38.48%.
Ash Content (AC)	27.50%
Volatile Matter (VM)	23.15%

(Akinlabi *et al* 2005)

The Ash content indicates the presence of elements in the rubber latex. Ash is what remains when water and organic materials are removed by heating the latex which is about 27.50% of the quantity measured as indicated above in Table 1. Volatile matter is the measure of heat removable matter in a sample and it's about 23.15 % of unwanted materials in the rubber latex.

The XRF analysis showed that the chemically modified NRL had higher concentration of the chemicals used (about 620 ppm) while the unmodified NRL did not shown traces of such chemicals at all. A typical XRF results of NRL and epoxidized natural rubber latex (ENRL) is as shown below:

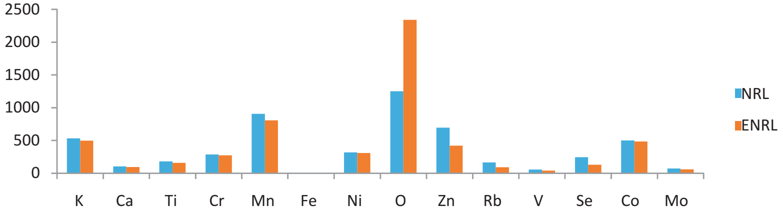


Fig 5 XRF result for natural rubber latex compared with epoxidized rubber**(Akinlabi *et al* 2011)**

It will be observed that the value of oxygen in NRL was lower when compared with that of epoxidized natural rubber latex. The effect of oxygen used in epoxidation had reflected in the results. Generally, most of the XRF results obtained do reflect the presence of the modified chemicals in the final results when compared with the unmodified.

The Fourier Transform Infrared (FT-IR) spectroscopy results revealed the presence of the functional groups before and after modifications: Basically for NRL and ENRL, the following functional groups are always observed: O?H (3439cm^{-1}) for modified and unmodified NRL, O?H (3450cm^{-1}), a broad band which is due to the presence of free O?H and intermolecular hydrogen bonds; C?H stretching (2931cm^{-1}) for modified and for unmodified (2925cm^{-1}), C?O stretching for the modified

(1182cm^{-1}) which is slightly higher than that of unmodified (1052cm^{-1}), C=O stretch for both (1642cm^{-1}), C=C (1612cm^{-1}) for both. There was an increased absorbance in the modified sample possibly as a result of epoxidation signifying that there could be a stronger hydrogen bonding for the interaction of the rubber matrix. A typical example of the FT-IR is as shown in Fig. 6.

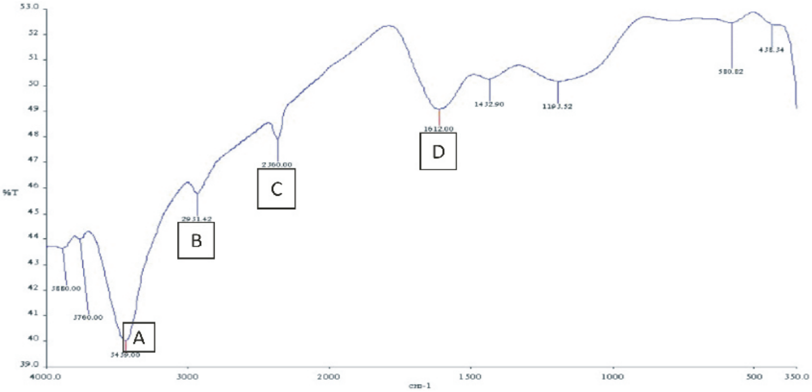


Fig. 6FT-IR spectrum of modified natural rubber latex (Akinlabi *et. al.* 2011).

Despite the wide range of the properties of NR, it still has some shortcomings that affect its application. For example, NR is susceptible to breakdown by oxidative and thermal degradation due to an unsaturated backbone, especially when it is presented in virgin form without any modification.NR latex also does not meet the requirements of some stringent industrial applications as a result of poor resistance to chemicals, problems of coagulation, the need for preservation, concentration etc. The above disadvantages have limited widespread applications of NR. However, I have made great efforts to improve these limitations of NR, which have now made NR to be available in different forms, easily transported without degradation, able to be preserved for longer periods and still retain its properties, increased its

susceptibility to oxidative and thermal degradation through oxirane ring formation. We have published 8 articles in this area: (Akinlabi *et al,* 2001, Aigbodion *et al,* 2001, Okieimen and Akinlabi 2002, Egbon and Akinlabi 2003, Akinlabi and Egbon 2003, Akinlabi *et al,* 2004a, Akinlabi *et al,* 2004b, Akinlabi *et al,* 2005a, Akinlabi *et al,* 2005b, Asia *et al* 2007)
Generally, my findings on chemical modifications has produced series of publicationsamong which are: Akinlabi *et. al,* 2003b, Akinlabi *et. al,* 2004a, Akinlabi . *al,* 2004b, Akinlabi *et. al,* 2005, Asia. e*t. al.* 2007. We were able to conclude that natural rubber latex can be modified chemically via all the processes mentioned and the extent of chemical modification affects the molecular weight and processing characteristics of the polymer. This is an indication that the modified NRL is expected to have high gel content and confer greater rigidity and stronger interaction with additive in polymer due to the interfacial matrix resulting from good dispersion of the particle.

(iii) Solving problems of low resilience high molecular weight natural rubber

These problems have militated against the use of NR in some products. The reduction of the molecular weight of NR was considered as a factor to help in facilitating the processing of the NR for some products. Production of liquid natural rubber and low molecular weight natural rubber of various degrees have greatly solved these problems. In the year 2000, Prof Felix Okieimen and I commenced a study on this and we were able to produce liquid natural rubber and low molecular weight natural rubber by depolymerization methods. We have been able to published the outcomes in local and international journals with several authors now referencing our work. The articles in production of low molecular weight natural rubber of various grades have provided another new area for rubber utilization, which is now gaining commercial acceptability **(**Akinlabi *et. al,* 2005a, b and c, Malomo *et. al.,* 2005,Ehabe *et. al.,* 2005)

(a) Development in Liquid Natural Rubber Production

Natural rubber's origin could lead to belief that it would have higher purity than synthetic rubbers. However, although it has no catalyst or monomer residue (as is the case with synthetic rubbers) natural rubber contains several impurities, e.g. proteins, which have negative impacts in some areas of application. One of the negative effects is the yellowish-brown colour that can be seen in some cases, e.g. baby bottle teats and medical devices. Odour, allergic reactions, lack of consistency and especially in sensitive applications are subjected to stringent regulations and therefore require high purity raw materials. The limitations in the scope of application of natural rubber are made more severe by some of its inherent properties viz:

- poor resistance to oils and hydrocarbon solvents
- high permeability to gases
- high sensitivity to ozone attack, etc.

Because many of these drawbacks can be alleviated by chemical modification based on the reactivity of the olefinic unsaturation in natural rubber, there is considerable interest in natural rubber modification.

Our interest in the modification of natural rubber is through the addition reactions of the olefinic unsaturation in the polymer to produce new and/or novel materials and/or intermediate materials that would expand the scope of application of natural rubber. It was conceived that epoxidation reaction may satisfy the research strategy. Thus we started off by investigating the epoxidation of crumb natural rubber and natural rubber latex to obtain data on the reaction conditions and other parameters that would be useful in monitoring the extent of modification and examined the tensile mechanical properties of blends of epoxidized natural rubber and natural rubber. We found that although higher levels of epoxidation may be attained with natural rubber in the latex form, large amount of surfactant are needed to be added to the latex concentrates inorder to delay/prevent coagulation. We then treated the epoxidized natural rubber with thioglycolic acid in the hope that the incorporation of thiols groups would lead to the production of self-curing rubber. Both the epoxidized natural rubber and its reaction product with thioglycollic acid were characterized by chemical and spectroscopic methods. Although the thiolated, epoxidised natural rubber was not self-curing, it appeared that we literally stumbled into novel rubber materials with interesting properties. We subsequently examined the physio-mechanical properties of blends of natural rubber and the modified rubbers.

Generally, reactions of crumb natural rubber in solution are limited by high viscosity. Even at low concentration (about 1%) solution of crumb rubber has sufficient high viscosity that may affect reaction process, thus optimum conversions were not attained. One strategy to improve on the extents of modification was the attempt to reduce the molecular weight of natural rubber and therefore the viscosity of the solution. We took advantage of a peptisation process employed in the pretreatment of natural rubber, prior to the determination of some of the raw rubber properties, such as plasticity rentention index. The peptisation process causes random chain scission of the polymer.We carried out investigations on the chain scission events associated with the treatment of natural rubber with nitrobenzene in the presence of ultraviolet light (sunlight).We found that the molecular weight of natural rubber could be reduced by up to 5-fold within two hours of reaction (Akinlabi*et al*2000). Further studies showed that 10-fold reduction in molecular weights of natural rubber are attainable see Table 2 (Okieimen and Akinlabi 2002).
These low molecular weight versions of natural rubber are referred to as liquid natural rubbers. The significant advantages of liquid rubbers are their ability to vulcanize with conventional networks and to improve processing and loading of the uncured network (Tables 3, 4 and 5). These advantages translate into the

following technical benefits (Okieimen and Akinlabi 2002, Akinlabi. *et. al.,* 2007a and b,Malomo *et. al.,* 2007, Malomo *et. al.,* 2010).

- Higher loading
- Reduced scorching
- Lower energy requirement
- Improved mould flow
- Lower plasticity
- Non- volatile extraction
- Increased green tack

We have developed the capability to produce liquid natural rubber with pre–determined molecular weights to meet diverse end-use applications, such as in adhesives, glue tack, high impact multifunctional additives, intermediates for polymeric products,etc.

The physico-mechanical properties, solvent resistance and transport properties (sorption, diffusion and permeability) of blends of liquid natural rubber and its epoxidised and thiolated derivtives with natural rubber have been studied (Okieimen & Akinlabi. 2002;Okieimen et al., 2003a&b; Akinlabi and Okieimen, 2004; Akinlabi *et. al*., 2005a,b&c). We summarized by remarking that liquid natural rubber is a versatile formation tool. It provides the unique combination of improved uncured batch processing and improved properties of the cure products. It continues to find use in creative formulation to develop new rubber compounds for innovative and demanding applications.

Reducing oxidation problems via depolymerization and reduction of chain length. – Reduction of the chain length and the number of double bonds in NRL was successfully carried out using nitrobenzene as the depolymerizing agent as shown in Table 2. Standard rubber recipe shown in Table 3 was adopted for compounding and results obtained shown in Table 4.

Table 2. Effect of Nitrobenzene concentrations on the extent of depolymerization of natural rubber latex. (Akinlabi *et. al.,*2007)

	Concentrations of nitrobenzene (%)				
Time(hr)	0.5	1	1.5	2	2.5
0	35,000	35,000	35,000	35,000	35,000
0.5	35,000	35,000	35,000	32,000	31,000
1	28,000	26,600	24,000	23,400	22,300
1.5	19,900	19,600	18,500	17,000	15,000
2	14,700	14,300	14,200	12,000	12,000
4	11,000	10,000	10,200	10,200	10,000
6	9,400	9,400	8,000	86,000	8,500
8	9,400	9,000	8,000	8,600	8,300
10	8,800	8,600	7,800	7,400	7,400

Table 3 Recipe for the preparation of NR / LMWNR blends(Akinlabi *et al* 2007)

Compound component	Samples (Pphr)								
	A	B	C	D	E	F	G	H	I
Natural rubber	100	95	90	75	50	25	10	5	-
LMWNR (26,600 Mw)	-	5	10	25	50	75	90	95	100
Carbon black (N330)	40	40	40	40	40	40	40	40	40
Zinc oxide (ZnO)	4	4	4	4	4	4	4	4	4
Sulphur	2	2	2	2	2	2	2	2	2
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
MBTS	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Flectol H	2	2	2	2	2	2	2	2	2

LMWNR = Low molecular weight natural rubber, MBTS= Dibenzothiazyl disulphide; MBTS = Dibenzothiazyl disulphide (MBTS)

Table 4 showing physico-mechanical properties of the rubber blends. (Akinlabi *et. al.*2007)

Physico-mechanical properties.	Mixes A	B	C	D	E	F	G	H	I
TS (MPa)	24.8	24.3	23.6	22.4	21	18.9	13.9	11.1	9.6
M50 (MPa)	2	1.8	1.8	1.7	1.5	1.4	1.2	1.2	1.1
M100 (MPa)	5.4	5	5	5	4.8	4.6	4.5	4.1	3.4
M300 (MPa)	8.0	7.9	7.7	7.7	7.4	7.1	6.8	6.5	5.7
EB (%)	880	889	912	918	923	923	900	894	699
Shear modulus (G)	0.424	0.435	0.439	0.441	0.443	0.441	0.439	0.431	0.428
CD x10^{-4}	1.6	1.6	1.6	1.5	1.5	1.5	1.4	1.4	1.4
Mc	5.5	5.6	5.6	5.6	5.4	5.4	5.3	5.2	5.1
V2	0.8	0.8	0.8	0.7	0.7	0.7	0.7	0.7	0.8
Hardness (IRHD)	58	58	56	55	52	50	48	46	42
Compression set	42	42	42	43	43	43	43	43	43
Abrasion resistance	61	61	60	58	56	56	52	49	46
Mooney viscosity	80	78	77	76	73	70	65	63	59

Table5 Calculation of intrinsic viscosity for average molecular weight.

Sample	Conc	$t_{s(s)}$	$t_{o\ (s)}$	t_s/t_o	t_s/t_o-1	**? SP/C**
A	0.02	300	285	1.053	0.053	250
	0.04	285	285	1.000	0.000	125
	0.06	300	285	1.052	0.053	83.3
	0.08	248	285	0.870	0.13	62.5
	0.10	300	285	1.053	0.053	50
B,	0.02	295	285	1.035	0.035	230
	0.04	327	285	1.147	0.147	115
	0.06	312	285	1.095	0.095	76.66
	0.08	294	285	1.032	0.032	57.50
	0.10	297	285	.042	0.042	46.00

Wherets is the viscosity of rubber , t_o is the viscosity of toluene, c is the concentration , ? is intrinsic viscosity. (Akinlabi *et.al.,*2007)

The intrinsic viscosity was determined from the graph plotted using the raw data shown in Table 5. Previously, Ulbelhode viscometer was used to determine the time taken for solution to flow. The graphs plotted were used to calculate the viscosity average molecular weight using mark Houwinks equation.

$M_V = K(?)^a$

Where;

M_v is viscosity average molecular weight;

? is intrinsic viscosity obtained from the graph and;

K and a are constant for rubber in specific solution. K= $1.50X10^4$ and a= 0.715

(b) Mixing schemes and relaxation models- life span behavior

The fact is that natural rubber on its own does not possess the required strength and crosslinking needed for polymeric material until sulphur is added. Addition of sulphur in rubber compounding is very important and crucial. I investigated the effect of various ways of adding sulphur in a standard rubber formulation as shown below in Table 6

Table6 Recipes for the four different vulcanization systems

Compound component	(Pphr)[a]	(Pphr)[b]	(Pphr)[c]	(Pphr)[d]
Natural rubber	70	70	70	70
LMWNR (50% reduction)	30	30	30	30
Zinc oxide (ZnO)	5	2	2	3
Stearic acid	0.7	1.5	1.5	1
Carbon black (HAF)	40	40	40	40
Sulphur	2.5	0.4	0.5	1.5
Flectol H (antioxidant)	2	2	2	2
MBTS	0	2.5	0	1.5
CBS	0.5	0	2.5	0
TMTM	0	0	1	0

[a]Conventional vulcanization system (CV), [b]Efficient vulcanization system 1 (EV1), [c]Efficient vulcanization system 2 (EV2), [d]Semi-efficients vulcanization system (semi-EV), Flectol H- Polymerized 1,2 dihydro-2,2,4-trimethyl quinolone, MBTS- Dibenzothiazyl disulphide, CBS- N-cyclohexylbenzothiazole-2-sulphenamide, TMTM- Tetramethylthiuram monosulphide.

(iv) Making natural rubber a multifunctional material

(a) Blending of natural rubber with synthetic polymers

There is the need to expand the scope of applications of natural rubber. In this regard, we decided to blend some polymers with natural rubber in anticipation of synergistic effects on the end products. We have blended natural rubber and modified natural rubber with other polymers like of poly(styrene), poly(propylene), poly(acrylonitrile), poly(methylmethacrylate), etc. The results obtained in each of the blends gave promising applications. The team and I have also published about seven articles in this field. The flexibility of natural rubber has been a major advantage in this area. We were able to detect that properties like mechanical, thermal stability, electrical conductivity, and so on were improved through the blends. Some of the intermediates obtained from the blends were found to have multi uses in that they combined the characteristic properties of the blended polymers. The following articles have been published in this area (Okeimen and Akinlabi 2003, Okieimen *et. al.,* 2003a&b, Akinlabi *et. al.,*2004, Aigbodion *et. al.,* 2005, Akinlabi *et. al.,* 2005, Akinlabi *et. al.,* 2007a,b&c).

(b) Treatment of waste water, effluents and oil spillage with modified natural rubber

Modified natural rubber was used in the removal of some heavy metals from effluents and waste water. The rubber was also recovered at the end of the experiment. We are now going further with one of my PhD students on this. He has designed several columns, packed them with different concentrations and different types of modified rubbers and additives. We had characterized all the effluents before and after passing through the columns packed with modified rubbers. We are left with combination of 2 or 3 columns to see if there would be any appreciable effect before we proceed toreusability and durability tests. This research has been found to be very interesting and promising. We are hopeful of getting it commercialized later for domestic usage. Some articles published in this areaare:Akinlabi and Koleoso 2014, Akinlabi *et. al.,* 2016, Alayande *et. al.,* 2017, Akinlabi *et. al.,* 2017b, Akinlabi *et. al.,* 2018

The release of crude oil and its derivative into marine ecosystem has caused adverse effect to aquatic life and this has led to environmental pollution. There is the need to protect the environment. Chitosan, a biodegradable agricultural waste from land snail shell, which is readily available in the market can be used as rubber additive for the clean-up of oil pollution and this can serve as a substitute to widely used imported rubber additives that attract high cost**.**

The study investigated the absorption efficiency of modified Natural Rubber Latex for the cleanup of oil pollution.Sorption efficiency of all the absorbents was determined by the gravimetric method. All the sorption tests were performed at 24 ± 2 °C. The efficiency of sorption of oil from water was carried out using gravimetric method reported by Swarnalatha *et al.,* 2012. The percentage sorption of oil was calculated using the equation below:

$$\text{Percentage sorption} = \frac{(Wt - Wo)}{Wo} x\ 100$$

Where W_o weight of the dry absorbent before immersion and W_t = weight of the sorbent after time 't' . The results are indicated below in the Tables 7, 8 and 9

Table 7 Petrol sorption data for all the sorbents

Sorbent	Percentage sorption after 1 h	Max sorption (g of oil per g of sorbent)
NR	259	8.91 (after 23 h)
NR-C	306	10.71 (after 23 h)
ENR-2h	435	7.86 (after 3 h)
ENR-C 2h	262	9.82 (after 23 h)
ENR-4h	265	6.32 (after 3 h)
ENR-C 4h	170	9.30 (after 26 h)
ENR-6h	75	1.75 (after 1 h)
ENR-C 6h	16	1.27 (after 1 h)

Table 8 Crude oil sorption data for all the sorbents

Sorbent	Percentage sorption after 1 h	Max sorption(g of oil per 0.2g of sorbent)
NR	120	1.75 (after 23 h)
NR-C	115	1.58 (after 23 h)
ENR-2h	150	2.15 (after 22 h)
ENR-C 2h	110	1.44 (after 23 h)
ENR-4h	125	1.69 (after 22 h)
ENR-C 4h	65	0.90 (after 22 h)
ENR-6h	90	0.44 (after 3 h)
ENR-C 6h	70	0.38 (after 3 h)

Table 9 ENR-C Adsorption isotherm parameter values

Isotherms	Parameter	ENR-C 2h	ENR-C 4h
Langmuir	Qm (mg/g)	0.8425	0.9102
	a	1.73510.6120	
	Rl	0.3417	0.1137
	R^2	0.9591	0.9552
Temkin	A_T (L/g)0.746	1.2305	
	B(J/mol)	-0.291	0.21
	R^2	0.8818	0.9274

Findings

This study has shown that modified natural rubber with chitosan from land snail shell can be deployed as a sorbent for oil spill treatment. The sorption and stability behavior of natural rubber, modified natural rubber were enhanced after chitosan incorporation. The sorption of oil occurred by surface reactions.The equilibrium sorption on modified natural rubber chitosan at 2 and 4 hours fitted the Langmuir adsorption isotherm having the highest regression value. The sorbents synthesized have good sorption capacities to be applied practically and few of the sorbents could be satisfactory reused. NR-C and ENR-C 2h were the suitable sorbents for sorption capacity of both petrol (gasoline) and crude oil, while NR-C, ENR-C 2h and ENR- 4 displayed a better reusability compared to other sorbents. Thus, the ENR and ENR-C demonstrated a potential for remediation of oil contaminated water.

(c) Synthesis of titanium (iv) oxide rubber composite membrane for photocatalytic degradation of synthetic dye.

The textile industry has been considered as one of the world's worst offenders in terms of pollution because 10-15% of all the dyes used in the industry are released to the environment with wastewater during processing. The presence of even very low concentrations of dyes in the effluent is highly visible and degradation products of these textile dyes are often carcinogenic. Hence the need to develop treatment techniques that can lead to complete destruction of the dye molecules from the waste stream. Adsorption is a useful and simple technique which has gained considerable attention in the recent years (Saad *et al*., 2007). However, the photocatalytic systems merely transfers the dye from one form of waste to another (*e.g.* liquid to solid) and therefore, cannot be considered to be a complete treatment of the waste.

Recently researchers have used photocatalytic approach capable of decolourising and degrading textile industry effluent in an effective way without leaving a trace of carcinogenic compounds. This approach is an advance oxidation technologies (AOT) for the treatment of these recalcitrant pollutants to more biodegradable molecules.

In the present study, natural crumb rubber was utilized as a support for TiO_2 nanoparticles by rheomixing. The objective was to synthesize TiO_2 nanoparticles natural rubber composite membrane for the degradation of synthetic dye using visible light irradiation under operational conditions such as pH, concentration and catalyst dose.

Synthesis of Nanoparticles

TiO_2, Ag-TiO_2 and Ag-Zn-TiO_2 nanopowders were prepared via sol-gel method using the precursor titanium tetraisopropoxide (TTIP, 97%, Sigma Aldrich), deionized water and 2-propanol (99%, Sigma Aldrich) as the starting materials (Perumal *et al*., 2014).

Preparation of Natural Rubber Composites

Twenty (20) g of dry crumb rubber was mixed with a required amount of undoped nanoTiO_2, doped or codoped TiO_2 nanoparticle as calculated by weight percent (wt%) based on the amount of the crumb rubber used in Thermo Scientific Haake PolyLab OS

RheoDrive 7 connected to Rheomixer. The RheoDrive 7 was connected to Polysoft OS software where operation conditions were set (mixing temperature, 200°C and time, 20 min). After mixing, the mixer was disconnected and the extruder fixed to the RheoDrive 7 and the compressor was switched on. The operation conditions were reset on the Polysoft OS software and the mix was loaded into the extruder. The composites were extruded out in a flat shape 40cm long.

Findings

The results of Brunauer-Emmett-Teller (BET) specific surface area values for undoped, doped and co-doped samples are shown in Figure 7. All the samples possess quite low specific surface area values, ranging from about 4.7 to11.8 m^2g^{-1}. The pore size is around 18.9-56.6 nm for all of the samples. The increase in surface area and pore size are attributed to the incorporation of dopant into the TiO_2 lattice. But the effect is more pronounced in 1 mol% Ag and 0.6 mol% Zn co-doped TiO_2 nanoparticle. The methyl orange dye adsorption is improved by doping as a consequence of increased pore volume and pore size.

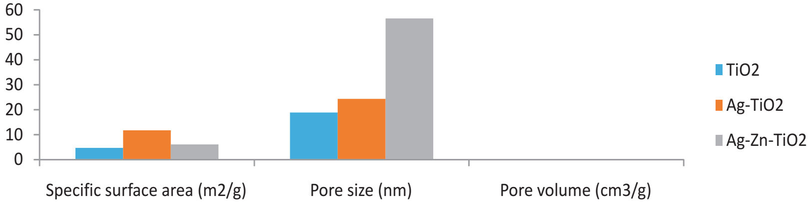


Fig 7 Specific surface area, pore volume, and pore size of undoped TiO_2, Ag doped, Ag and Zn co-doped TiO_2 nanoparticles. Note that the pore volume varies from 0.02, 0.07 and 0.09 for TiO_2, Ag-TiO_2 and Ag-Zn-TiO_2 respectively. (Akinlabi *et al* 2019)

The investigation on the adsorption of methyl orange on natural rubber composites filled with 4 and 6% nanoparticle under solar irradiation at pH 4.1 for initial dye concentration (4 ppm) showed that the composite filled with 6% Ag-Zn-TiO_2 nanoparticle has the highest adsorption and/or degradation of methyl orange at 33%, whereas 17 and 26 % were obtained for TiO_2 and Ag-TiO_2 respectively. However, 14, 25 and 21% degradation were obtained for composites filled with 4% TiO_2, Ag-TiO_2 and Ag-Zn-TiO_2 nanoparticle respectively. The lowest adsorption (8%) was observed for natural rubber without a catalyst. The low concentration of catalysts in the entire composite was responsible for the lower percentage photodegradation recorded in comparison of photodegradation of nanoparticles as indicated in Figures 8 and 9.

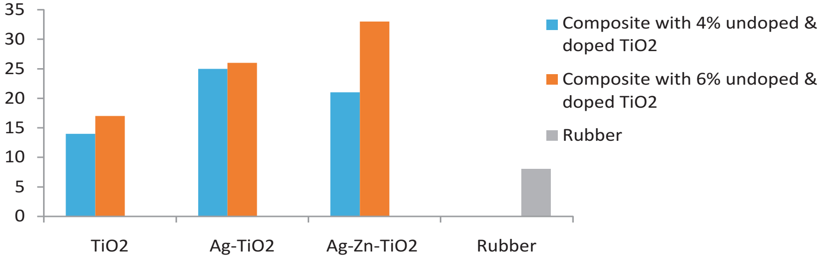


Figure 8 Photodegradation of methyl orange dye by composite filled with or without catalyst at pH 4.1 (Akinlabi *et. al.,* 2019)

The apparent-first-order rate constant for the natural rubber composites filled with nanoparticles is also monitored at pH 4.1. The composites filled with 4 and 6% TiO_2 nanoparticle seemed to show poor photodegradation and their apparent-first-order rate constants are approximately zero. The highest photodegradation rate is observed at 0.00182 min^{-1} for composite filled with 6% Ag-Zn-TiO_2 nanoparticle followed by composite filled with 4% Ag-TiO_2 nanoparticle (0.00076 min^{-1}). The composites filled with 4% Ag-Zn-TiO_2 and 6% Ag-TiO_2 nanoparticle hadapparent-first-order rate constant of 0.00054 and 0.00065 min^{-1} respectively. This simply revealed the rate at which the dye is degraded and consequently, the highest apparent-first-order rate constant gives the highest percentage degradation

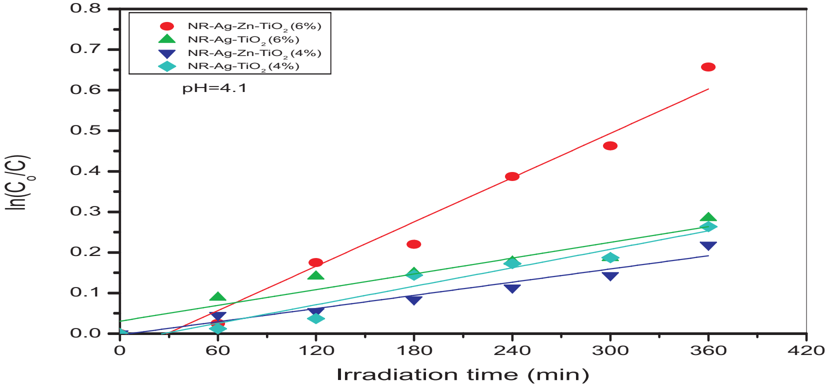


Figure 9 Effect of composites on Kinetics of photodegradation of methyl orange dye at pH 4.1. (Akinlabi *et. al.,* 2019)

Findings

The study showed the effects of metal dopants on TiO_2 nanoparticle and subsequent immobilization on natural rubber for photodegradation of methyl orange dye as a model organic pollutant under solar simulator. A single phase undoped, doped or co-doped rutile TiO_2 nanoparticle is synthesized by sol-gel method. The dopants are found to increase the surface area and pore size of TiO_2 nanoparticle, and consequently enhance photocatalysis of NR-Ag-TiO_2 and NR-Ag-Zn-TiO_2 under the visible light irradiation.

(v) Natural rubber in drug delivery system

Natural rubber latex contains protein and carbohydrate in addition to other materials. In pharmaceutical industries, carbohydrates are used as additives for drugs while proteins are also used in treating some ailments e.g insulin is a protein for treating diabetes. It was in line with this that we decided to get natural rubber and liquid natural rubber modified and centrifuged for **GEL** preparation with the view of replacing the protein and modifying the carbohydrate. Centrifuging of the rubbers were done using stainless tubes with 12,000 revolution per minute. The centrifuged gels were subjected to analysis including rheometry, Mass spectometry coupled with Gas chromatography and viscometry analysis. Summarily, the results showed that the carbohydraes can also find uses in drug delivery while the protein was replaced with insulin for potential uses in treating diabetic patients. The quantity of protein and carbohydrate obtained were very small and we are currently working on how this can be increased. I have threepostgraduate students working on this and Dr Ilesanmi Nathaniel defended his PhD thesis on the use of modified rubber in slow release systems

(a) Using modified rubber in slow release of phytochemicals- The improvement in the style of drug release and increase in the demand for biomaterials that continuously release substances of pharmacological interest is on the increase in medicinal chemistry. This study developed an exceptional matrix-forming feature and sustainable system of delivery of insulins and phytochemicals with natural rubber latex matrix serving as the base for the transdermal patches. Natural rubber latex was centrifuged at 8000 rpm and deprotenized to remove the leachable protein. The modified natural rubber latex and bioactive compound (phytochemicals and insulins) were polymerised at 10, 15, 35, 50 ˚C for phytochemicals and 15, 30, 40 and 50 ˚C for insulins. The biomembrane was characterized using Scanning Electron Microscopy (SEM), Fourier Transform Infrared (FTIR), Ultraviolet-Visible Spectroscopy, X-Ray Diffraction and ImageJ.

Findings from the research has pictured in Figures 10 to 12 showed that it is possible to produce transdermal patch with low extractable protein contents and low allergic responses from re-centrifuged latex at a very high revolution of 8000 rpm. We achieved a 52.25% reduction in protein content of natural rubber latex caused by hydrolysis of the high molecular weight protein by alkaline solution. The research also confirmed that ammonia does not only prevent coagulation of rubber latex but also help in deproteinisation of natural rubber latex.

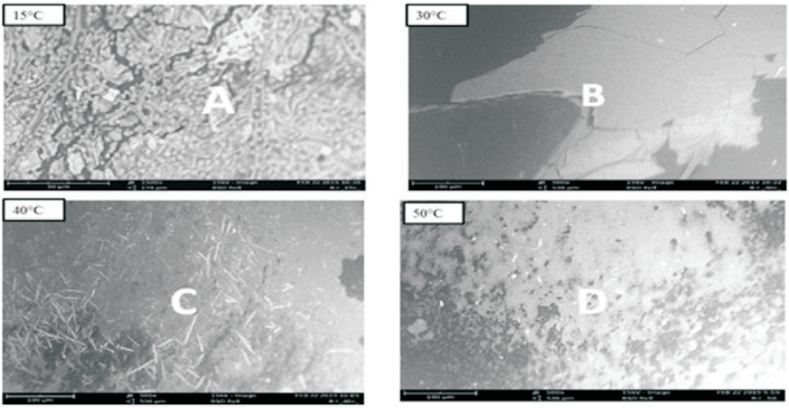


Fig. 10SEM micrographs of insulin biomembrane polymerised at different temperature (Akinlabi *et. al.,* 2019)

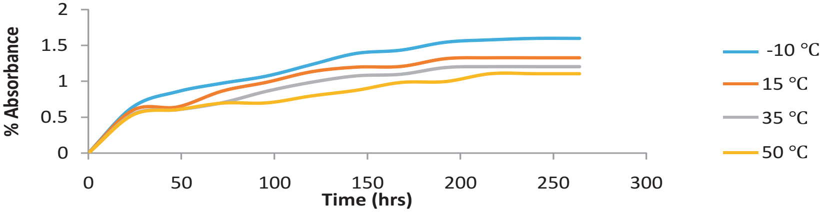


Fig. 11Release pattern of phytochemical from phytochemical biomembrane polymerized at different temperatures(Akinlabi *et. al.,* 2019)

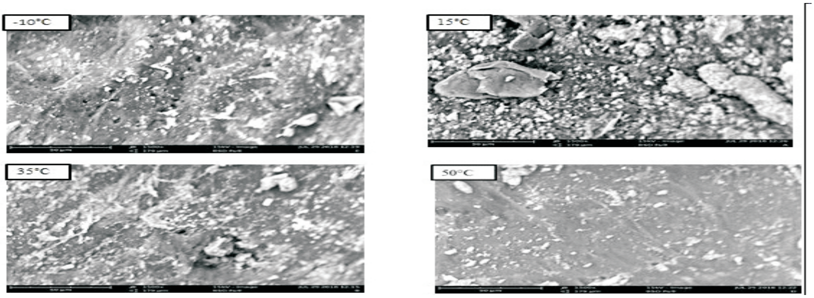


Fig. 12 SEM image of phytochemical biomembrane polymerized at different temperatures

(b) Using modified rubber as transdermal patches-
We had a thought on plaster bandage for external application of medicine on skin. External applications of drugs in form of creams, oils, plaster, pomades are frequently applied and we thought of impregenating our modified natural rubber with the active component of some drugs (anti-fungal and anti-dermal extracts) so that the sticky property of rubber can be exploited as a way of plastering it on the affected part and releasing the drug potent gradually over a period of time. This has been tried on rats (Figures 13 – 16) and we are still on it with the expectation of getting positive results. We have done the in-vivo test and will do the in-vitro tests.

Fig. 13 Shaving of the rat

Fig. 14 Infection of the rat

Fig. 15 Application of the modified NR

Fig. 16 Healing of the affected parts

(a) Modified rubber with Baobab extracts

Another plant that is currently under investigation for its drug potencies is Baobab. Baobab is the fruit of Africa's 'Tree of Life'. It is the only fruit in the world that dries naturally on the branch. Baking in the sun for 6 months, baobab's green, prickly, velvet-like coating transforms into a smooth, brown, coconut-like shell. The hard casingcan be cracked with the use of a hammer. The inside has a dry whitish pulp of the fruit which contains no moisture whatsoever. Baobab powder is a rich source of vitamin C, which contributes to normal energy release, Immune function and healthy, glowing skin. Baobab is also almost 50% fibre (two thirds soluble and one third insoluble, making it a powerful prebiotic) and contains more antioxidants than any other whole fruit (Addy *et al* 1995). Baobab is more bioavailable than manufactured vitamin supplements, meaning our bodies can absorb the nutrients more easily, ensuring a greater uptake. In addition, amazingly, baobab has a natural shelf-life of 3 years so there are no additives or preservatives needed.The idea was to investigate the medicinal application of baobab extracts as antifungi and antidermal and possibly blend it with natural rubber latex to form a thin film and study the rate at which the active component of the baobab will be released from the rubber film. The following articles have been published in the medical and drug aspects of our research (Adeseluka *et. al*., 2017, Akinlabi A. K. *et. al*., 2018a&b, Kareem *et. al*., 2019, Oladoyinbo *et. al*., 2019,Oni *et. al*., 2020).

Fig.17 Baobab fruits

Fig. 18 Baobab seeds

Potential uses of baobab

The baobab fruit shown in Figures 17 and 18 was harvested and divided into 4 parts: the fibre, the powder, the pulp and the seeds. The seed was further processed to extract oil from it. The following tests were carried out on each of the four parts: Moisture Content, Ash Content, pH, crude fats, Volatile Matter, carbohydrate content, Nitrogen Content. All the four (4) parts were further treated for their potential utilization. It was found that baobab pulp has a huge potential use in medicine,food and will serve as a viable raw material for industries.

- *Uses in phytoremediation of heavy metals in water*

Baobab fruit fibre was used to absorb chromium, cadmium, zinc and lead in well water from kuto, Lafenwa and crescent university as shown in Figure 19. Micro pixe analysis and FTIR analysis of the baobab fibre before and after absorbtion were recorded. Atomic Absorption Spectrometer (AAS) analysis of the water from the three different locations (kuto, lafenwa and crescent) before and after the treatment was also conducted. Results showed that Lafenwa water was contaminated more with Pb followed by Zn, Cr and then Cd same as that of Kuto. Crescent water was not found to be contaminated at all. The results presented in Figure 19 showed that the fibre has the ability to retain Cd, Cr, Zn and Pb by absorption and graphically shown below, which confirms that baobab fruit fibre can be used in phytoremediation of heavy metals in soil and water.

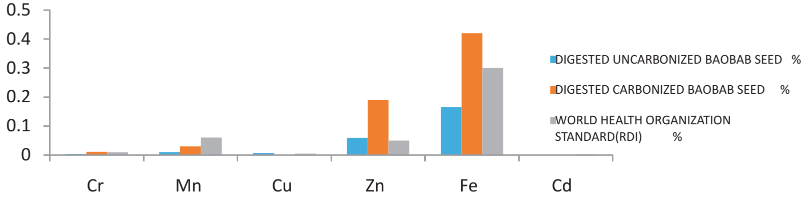


Fig. 19 Effect of Baobab seeds on phytoremediation of some metals

- *Phytochemicals in Baobab*- The phytochemical screening of the biomembrane polymerized with *adansonia digitata* extract showed that alkaloids, tannins, saponins, terpenes, flavonoids, phlobatanins and steroids were present in ethanol extracts but anthraquinones was absent in the extract. Tannins, saponins, alkaloids, phlobatannins, steroids, and flavonoids have been suggested to be involved in anti-bacterial and anti-viral activities, while tannins and flavonoids are thought to be responsible for anti-diarrheal activities.
- *Transdermal uses*-For any material to serve as a transdermal patch, the material must have the ability to accommodate active ingredient in its structure and remain unreactive with the compound. FTIR spectra also revealed that there was no chemical interaction between the bioactive compound and the NRL membrane; however, the bioactive compounds were adsorbed on the polymeric matrix. The images from the SEM showed a bioactive portion on the surface of the matrix, which was responsible for the fast release..
- *Antioxidant uses*- The antioxidant activities of the bioactive substances were sustained even when they have been integrated into the matrix. The results confirmed that the biomembranes produced have shown effectiveness as a model in the release of bioactive compounds (phytochemicals and insulins) for transdermal purposes
- *Extraction of oil from baobab seeds*- Soxhlet extractor evaporator to obtain oil. The oil was stored in a labeled sample bottle. During extraction, temperature was maintained at 70 ^{0}C ± 2 ^{0}C and solvent to powder ratio at 1:3 (g/mL). The result of quality parameters of baobab seed oil obtained using different organic solvents extraction techniques are shown in Table 13.

Peroxide value measures the degree of oxidation of the unsaturated fatty acids This shows that baobab seed oil extracted with n-hexane had a higher peroxide value ranges from 24.00 ±

5.66 mEqKOH/kg in oil extracted with acetone and ethanol to 25.00 ± 7.07 mEqKOH/kg in ethyl acetate
extracted with n-hexane had a higher peroxide value ranges from 24.00 ± 5.66 mEqKOH/kg in oil extracted with acetone and ethanol to 25.00 ± 7.07 mEqKOH/kg in ethyl acetate

Table 10: Mean values for quality parameters of baobab seed oil

Solvent	PV (mEqKOH/kg)	IV (g/100g)	FFA (% oleic acid)	SV (mg/100g)	AV (mg/100g)
Ethyl Acetate	25.00a ± 7.07	93.25a ± 71.84	9.87a ± 1.99	107.95a ± 1.93	19.90a ± 1.41
N-Hexane	42.00a ± 2.82	87.67a ± 71.77	7.76a ± 0.99	251.33b ± 41.2	15.62a ± 0.98
Acetone	24.00a ± 5.66	84.72a ± 35.85	4.94ab ± 1.0	223.70b ± 1.00	9.65b ± 0.96
Ethanol	24.00b ± 5.66	89.42a ± 35.92	5.64b ± 0.00	209.55b ± 2.33	11.04c ± 0.14

Means with different superscript are significantly different ($p< 0.05$) PV represents Peroxide Value, IV represents Iodine Value, FFA represents Free Fatty Acid SV represents Saponification Value, AV represents Acid Values.

Results obtained from baobab oil characterization are presented in Table 10. Knowledge of iodine value enables the combustion temperature of oil to be evaluated. It also determines the ability of oil to form solid film when exposed to air. The iodine values obtained in this study suggests that it can be used in alkyd resin production, biodiesel production and other products formulations such as liquid soap, shoe polish, cosmetics,etc.

Saponification value is defined as the amount of potassium hydroxide (KOH) in milligrams required to saponify one gram of fat or oil. Oils with high saponification values would be less sensitive to deterioration (Nyakudya *et* al., 2015). Values obtained in this study as indicated on Table 9 were 107.95 ± 1.93, 251.33 ± 41.26, and 223.70 ± 1.00 and 209.55 ± 2.33 mgKOH/g for Ethyl acetate, n-hexane, acetone and ethanol respectively. There is no significant difference ($p < 0.05$) in all the extraction solvents except ethyl acetate that was significantly different, and this significant difference could be attributed to the physicochemical properties of the solvent. The baobab seed oil obtained from n-hexane had the highest saponification value as such, would contain more short-chain fatty acids for stability during storage. The baobab oils extracted with ethyl acetate had the lowest saponification values. This indicates the predominance of long chain fatty acids in these oils suitable for the food and cosmetics industries and for biodiesel production. the high saponification value of baobab seed oil indicates the high potential for its use as lubricant in polymer additives.

Free fatty acid is an index use in determining the quality of oils. The lower the free fatty acid content of an oil sample, the more desirable it will be as edible oil. It is recommended that edible oil should have free fatty acid values of less than 5 %. As indicated in Table 9, Free Fatty Acid values ranges from 4.94 ± 1.00 to 9.87 ± 1.99 % in acetone and ethyl acetate respectively. The values were in same range with that reported by Auquo(2010) and Ibiam and Anosika(2014) for Palm Kernel Oil and coconut oil. The low Free Fatty Acid level of baobab seed oil is associated with its high degree of unsaturation which makes safe as edible oil.

Acid value is the amount of Free Fatty Acid present in fat as measured by the milligram of Potassium Hydroxide needed to neutralize it. It reports the degradation state of oil by evaluating the amount of free fatty acids formed during extraction or storage. The acid values were between 9.65 and 19.90 mg/100g with oil extracted with Acetone having the lowest value which reveals the weakest acid level, therefore less sensitive to rancidity during extraction. Consequently, the hydrolysis of the ester bonds of the triglycerides is greater in the oil resulting from Ethyl acetate which has the highest value. The acid value, as shown on the table varies significantly ($p < 0.05$) among extracting solvents except for ethyl acetate and n-hexane which showed no significant difference. The overall result affirms the edibility of baobab seed oil just like coconut and palm kernel oil.

(vi) Local sourcing of additives for natural rubber compounding

(a) Utilization of clays and cement klin dusts,

The use of clays as additives in some polymeric materials has assisted in cost reduction and properties modification. Silicas, clays, termitarial soils and cement klin dusts have been investigated by my team as additives and they were found to have improvedthe physical properties of rubber vulcanizates developed from them. Akinlabi *et al.,* 2009 were able to discover that addition of any particulate material will reduce some of the physical parameters of a polymer more or less in proportion to the volume present. The only material of an exception to this is carbon black in rubbers and fibrous fillers. My team decided to look into clays of various compositions and different termitarial soils.Dr Gabriel Oladipo, Dr Kehinde Oderinde, Dr. Femi Falope, Mr Femi Oguntubo and Mr Biodun Mosaku have worked in these areas. We observed that whenever the volume loading of fibrous filler was high, the length-to-diameter ratios will also be sufficiently large to make the fibres themselves strong and act as reinforcing. This was later found to influence the tensile strength, stiffness, hardness and flexural strength since they were determined almost wholly by the fibre. We have produced seven papers on this. (Oladoja & Akinlabi 2009, Akinlabi *et. al.,* 2011, Akinlabi *et. al.,* 2013, Alayande *et. al.,* 2013, Akinlabi *et. al.,* 2015, Yaya and Akinlabi 2015)

(b) Using banana pseudostem fibres and calcium carbonate

Crumb rubber produced from natural rubber latex was compounded by using modified and unmodified banana pseudostem fibres with calcium carbonate. The banana pseudostem was first modified via sulphonation, hydroxylation, carbonization and nitration. The unmodified and modified banana pseudostem fibres (BPFs) were characterized via loss on ignition, elemental composition with X-ray Fluorescence (XRF), ash, volatile matter, crystallinity and particle size with X-ray diffractometer (XRD) while Fourier Transform Infrared (FTIR) was used for the functional group analysis. The crumb rubber was then compounded on the two roll mill machine with the modified, unmodified and calcium carbonate using the natural rubber standard compounding formulations. The vulcanizates obtained were cured at 150ºC for 5 minutes at a pressure of 260 psi to give test pieces used for physico-mechanical and chemical tests. The physico-mechanical tests showed that calcium carbonate samples gave the highest tensile strength, followed by the modified fibre samples while the unmodified fibre samples gave the least strengths. Within the modified samples, carbonization was found better than sulphonation, which was better than hydroxylation and better than nitration. Chemical tests were through swelling and solubility tests, which were carried out at room temperature in different solvents; acetone, petrol, diesel, kerosene and water. The swelling results showed that acetone swell the vulcanizates more than petroleum fuels (petrol ? kerosene ? diesel) and swell more than water. In summary, it was observed that the surface modification of the fibres increased the physico-mechanical properties of the vulcanisates especially when compared with unmodified vulcanisates.

Preparation of the Banana Pseudostem Fiber (BPF)

Banana pseudostems were retted to obtain the fiber and dried under the sun for three days after which it was transferred into a drying cabinet. The oven dried pseudostem fiber was ground using a hammer mill, into powder. It was then screened using a 100µm mesh. The fibers were kept in a desiccator before use.

Characterization of Banana Pseudostem Fibres

BPF was characterized using the methods stated by Akinlabi *et* *al.*,(2015). The following tests were carried out to ascertain its utilization as fillers in rubber compounding and these include moisture content, ash content, loss on ignition and pH of aqueous slurry.

*Surface modifications of Banana Pseudostem Fiber (BPF)*were conductedvia sulphonation, nitration, hydroxylation and carbonization

Recipe for the compounding

Standard formulation for modified natural rubber compounding mix was adopted. A typical formulation is as given in Table 11 below:

Table 11Compounding recipe for natural rubberblends(Akinlabi *et. al.,* 2005)

Mix Recipe	**A**	**B**	**C**	**D**	**E**	**F**
Crumb rubber (g)	100	100	100	100	100	100
Stearic acid (g)	2	2	2	2	2	2
Zinc Oxide (g)	5	5	5	5	5	5
TMTD (g)	1	1	1	1	1	1
Silica	20	20	20	20	20	20
Processing oil (ml)	3	3	3	3	3	3
Sulphur (g)	3	3	3	3	3	3
Sulphonated fibre	20	-	-	-	-	-
Carbonized fibre	-	20	-	-	-	-
Hydroxylated fibre	-	-	20	-	-	-
Nitrated fibre	-	-	-	20	-	-
Unmodified fibre	-	-	-	-	20	-
Calcium carbonate	-	-	-	-	-	20

A**:**Sulphonated vulcanisate B: Carbonized vulcanisate C:Hydroxylated vulcanisate D: Nitrated vulcanisate E: Unmodified vulcanisate F: $CaCO_3$

Findings

Characteristics of the banana pseudostem fiber (BPF)

The ash content of the banana fibre used in this study was 5.08%. Ash present in lignocellulosics contain silica that has many undesirable effects. The moisture content of the fibre was 9.52%. The determination of moisture content is very important because fibre dimension and properties vary with it. High moisture absorption of natural fibres leads to swelling and presence of voids which results in poor mechanical properties and reduces dimensional stability of composites. The value of the loss on Ignition of 54.54% indicates the carbon content present in the fibre. It shows the suitability of the fibre in crosslinking. The higher value of loss on ignition indicates the ability of the fibre to give a high level of reinforcement.

On the nitrated banana fibre, functional groups that were present are: N-H stretch for 1^o and 2^o amines, amides at 3377.65cm^{-1} , C-H stretch of alkanes at 2917.84cm^{-1} , $C\equiv N$ stretch of nitriles at 2227.15cm^{-1} , $-C\equiv C-$ stretch of alkynes at 2029.15cm^{-1} , N-O asymmetric stretch of nitro compounds at 1540.10cm^{-1} , C-N stretch of aromatics at 1161.07cm^{-1} and =CH-H bend of alkenes at 899.28cm^{-1}. On the unmodified fibre, the peaks present include N-H stretch for 1^o and 2^o amines, amides and O-H bond stretch of alcohols and phenols at 3344.83cm^{-1}, C-H stretch of alkanes at 2920.91cm^{-1}, $-C\equiv N$ stretch of nitriles at 2352.58cm^{-1}, C-H bend of alkanes at 1424.89cm^{-1}, N-O asymmetric stretch of nitro compounds at 1324.01cm^{-1} , C-N stretch of aromatic amines at 1161.66cm^{-1} and =CH-H bend of alkenes at 898.25cm^{-1}. The modification made by nitration did not make a significant change on the fibre. The nitrogen and hydroxyl groups seem to be the prominent functional groups whose sources could be from the fact that the banana is a lignocellulose fibre and the nitrogen could be from the uptake of nitrogen from te soil by the roots and the pseudostem since the pseudostem is closer to the soil as well as the root.

X-Ray Fluorescence

The X-Ray fluorescence results for the unmodified (Figure 20) and modified(Figure 21) fibres were examined to show the elements present and to confirm if the modification actually took place or not. For the unmodified BPF, Figure 20, the element with the highest concentration was potassium (8.4547 wt. %) which is followed by Iron (6.232 wt. %) and Calcium (5.0543 wt. %). The element with the least concentration is strontium (149 ppm). The presence of potassium as a Group 1 element makes its reactions with water vigorous due to its high concentration. It can also impose lighter weight on the fibre due to its low density. Calcium as an alkali earth metal is able to provide the fibre with lustre due to its high concentration and can impose hardness on the bulk. Iron as a transition metal possesses properties of hardness and high density same as other transition metals present which gives high tensile strength. Other elements such as strontium, arsenic and bromine are present in lower ppm but the presence of arsenic at such concentration of 1040ppm is not environmentally friendly as arsenic in lower concentrations pollutes the environment.

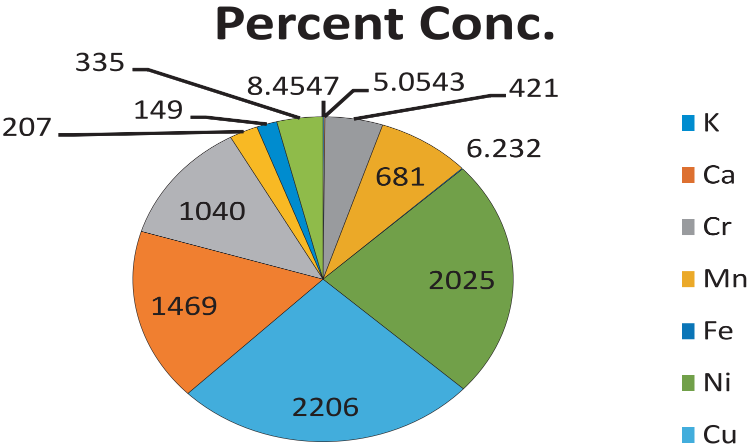


Fig. 20 Elemental composition of unmodified banana fibre(Akinlabi *et al* 2007)

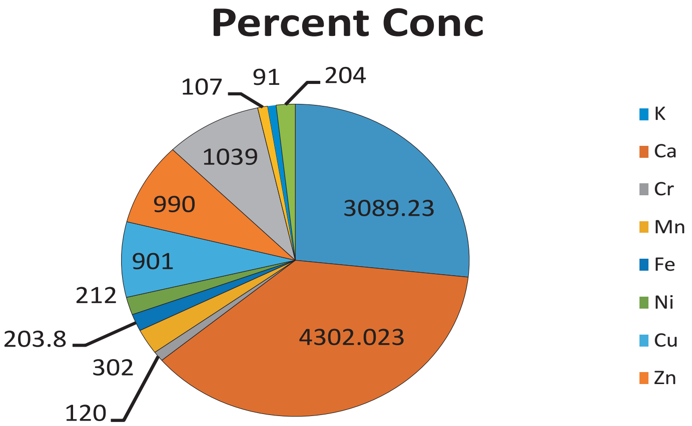


Fig. 21. Elemental composition of hydroxylated banana fibre(Akinlabi *et al* 2007)

For the hydroxylated fibre, Figure 21, the element with the highest concentration is Calcium (4302.023 ppm) which is followed by Potassium (3089.23 ppm) and then Arsenic (1039 ppm). The least element present in the hydroxylated fibre is Strontium (91 ppm). From the comparison between unmodified and hydroxylated fibre, it was noted that there was a reduction in the concentration of all the elements which indicated that the hydroxylated modification on the fibre gives a decrease in the concentration of all the elements present.

Physico- mechanical properties of the vulcanizates

In Figure 22, the tensile strength at yield load highest value was observed in carbonized vulcanisate with a value of 43.2 followed by $CaCO_3$ vulcanisate with a value of 40.5 and sulphonated vulcanisate with a value of 34.8, hydroxylated vulcanizate with a value of 30.3 while nitrated vulcanizate has the least value of 26.1. The highest value observed for carbonized vulcanisate was attributed to increase in the carbon content on the fibre, which was responsible for enhanced bond formation in the rubber matrix that gave rise to the high tensile strength. Tensile strengths are generally governed by filler dispersion, particle size, specific surface area of filler and rubber interaction (Muniandy *et al.*, 2002). The low value of tensile strength at yield load on the hydroxylated and nitrated vulcanisates could be as a result of poor dispersion of their fibres in the rubber matrix due to the acidic effects of hydrogen and nitrogen. Generally, acidity reduces network formation in rubbers (Egwaikhide *et al.*, 2007).

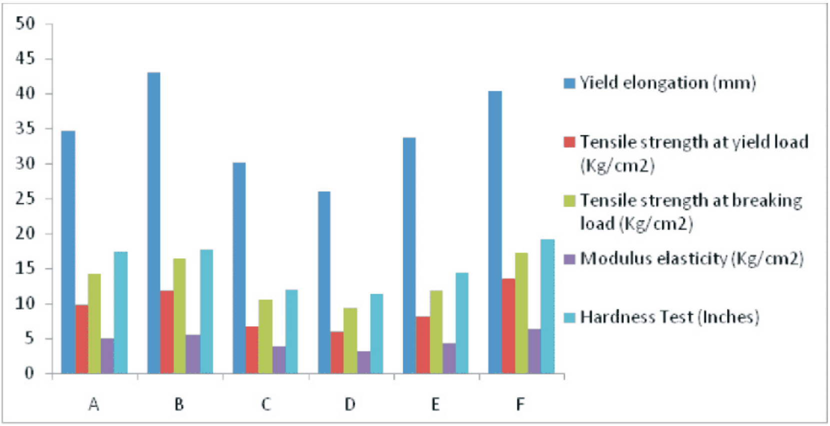


Fig. 22 : Physico- mechanical properties of vulcanisates(Akinlabi *et al* 2007)
A: Sulphonated fibre vulcanisate, B: Carbonized fibre vulcanisate C: Hydroxylated fibre vulcanisate D: Nitrated fibre vulcanisateE: Unmodified fibre vulcanisate F: $CaCO_3$ filled vulcanisate

In Figure 22, the tensile strength at breaking load was observed to be highest for mix F, ($CaCO_3$) vulcanisate with a value of 17.3 and followed by mix B with a value of 16.4, then mix A (14.2), mix E (11.9), mix C (10.5) and mix D with a value of 9.4 Kg/cm^2. From the tensile strength at yield load and the tensile strength at breaking load results, it can be deduced that $CaCO_3$ acted as reinforcing filler so also carbonization process assisted the fibres to reinforce the rubber vulcanizate. Sulphonation increased the tensile strength slightly while hydroxylation and nitration of the fibers did not show any positive reinforcing has reflected in their values as it was lower than that of the unmodified fiber. Hence, for banana pseudostem fibre, hydroxylation and nitration of the fiber might not be a good option to increase or improve the tensile strength.

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Elongation at % break is influenced by the adherence of the filler to the polymer phase leading to stiffening of the polymer chain and hence resistance to stretch when strain is applied. Material of higher elongation percent at break would be less rigid and would be expected to have low hysteresis and heat build-up. Unmodified vulcanisatesusually have high EB% when compared with the hydroxylated vulcanizate, which implies that hydroxylation reduces elongation at % break value. The vulcanisate with the highest value of elongation at % break will display a good toughening property which suggested why the dumb bell test pieces of unmodified vulcanisates did not snap easily on elongation.

Modulus is related to the stiffness and elasticity of vulcanisate. Chemical modification of the fibres will improve the surface area and surface reactivity of the fibres through filler dispersion and filler-rubber interaction thereby enhancing the elasticity of the vulcanisates. Hence, chemical modification has improved the elasticity of the vulcanizates as seen in Fig. 22. Hardness is the resistance to indentation and easily influenced by the compounding process, filler dispersion and distribution, which eventually affects the crosslink rate and density of the vulcanisate.

Chemical behaviour as shown by swelling and solubility results

The extent to which crosslinking has taken place in the vulcanizates was predetermined by the swelling and solubility tests. The vulcanizates were submerged in various solvents and the results obtained are as shown below in Table 12

Table 12 Swelling and solubility results of the vulcanisates in differentsolvents (Akinlabi *et al* 2010)

Solvent	Vulcanisates	W1	W2	W3	% Swelling	% Solubility
Acetone	1	0.5303	0.6402	0.4915	17.18	7.54
	2	0.4735	0.6020	0.4421	21.66	6.38
	3	0.4131	0.5630	0.3800	26.78	7.31
	4	0.4423	0.6121	0.4004	27.86	9.09
	5	0.4641	0.6431	0.4234	28.12	8.69
	6	0.4941	0.9004	0.4524	45.55	8.16
Petrol	1	0.3805	0.4583	0.3693	17.03	2.94
	2	0.3692	0.4494	0.3566	17.82	3.44
	3	0.3498	0.4323	0.3316	19.11	5.23
	4	0.4039	0.504	0.3823	19.90	5.31
	5	0.3598	0.4543	0.3226	20.82	10.32
	6	0.3476	0.4628	0.3065	24.93	11.83
Kerosene	1	0.4943	0.5737	0.4851	13.84	1.91
	2	0.4082	0.4816	0.3983	15.22	2.41
	3	0.4866	0.5777	0.4713	15.81	3.14
	4	0.3532	0.4300	0.3335	17.93	5.63
	5	0.3973	0.4858	0.3685	18.22	7.22
	6	0.3594	0.4429	0.3277	18.92	8.82
Diesel	1	0.4340	0.4506	0.3920	4.44	9.30
	2	0.4828	0.5129	0.4407	5.88	8.33
	3	0.4729	0.5038	0.4326	6.00	8.51
	4	0.4509	0.4814	0.4021	6.25	11.11

W_1 = is the weight after extraction. W_2 = is the weight after swelling at 25˚C for 48hrs. W_3 = is the weight after drying in a vacuum to a constant mass., 1: $CaCO_3$ filled vulcanisate, 2: Carbonized fibre vulcanisate, 3: Sulphonated fibre vulcanisate 4: Unmodified fibre vulcanisate 5: Hydroxylated fibre vulcanisate 6: Nitrated fibre vulcanisate

Solvents have different penetrating powers but the resistance of materials to such penetration greatly depends on the intermolecular arrangement. Scientifically, when intermolecular force of attraction is high, materials have tendencies to be highly crosslinked and higher forces will be required for molecular rearrangement, hence leading to difficulties in solvent penetration. Swelling result is also an indication of the extent of crosslinking and it shows the strength of the vulcanizates in terms of curing. The results in Table 12 generally shows that acetone penetrated more than petrol, more than kerosene, more than diesel and more than water. This was probably due to the differences in the solvents molecular masses and diffusion coefficiency coupled with the fact that kerosene being a mixture of hydrocarbon C_{10}– C_{14} has a lower molecular weight than diesel C_{14} – C_{19}. Hence, kerosene will be expected to diffuse faster than diesel.

Resistance of vulcanizates to solvents is usually enhanced by uniform distribution and dispersion of compounding ingredients in the rubber matrix, which leads to a well crosslinked vulcanizate. Looking at the behaviours of the mixes in various solvents, the swelling results follow a particular pattern in all the solvents with mix 1 having the least value and it increases to mix 6, having the highest value. The solubility results were almost following the swelling pattern apart from some deviations caused by some of the vulcanizates going into solution making it slightly difficult for computation. Swelling and solubility of natural rubber has been documented by Baker *et al* 1995 to be influenced by various factors, such as crosslink type and density, amount, nature and type of compounding ingredients and of elastomer. It is also worth mentioning that swelling of the vulcanizates for longer periods in acetone showed the vulcanizates turning into slurry and the samples became difficult to weigh. This could have been responsible from the prolonged penetrating power of the solvent thereby weakening the resistance of the vulcanizate and penetrating the vulcanizates through the segmental packing and crosslinked matrix. During solubility determinations, the weights recorded after re-drying in the vacuum chamber were lower than the original weight of the samples which can be due to some samples that might have possibly gone into solution, and this accounts for the solubility percentage measured and reported. Above all, the physico-mechanical, swelling and solubility results obtained for the mixes were found to be within the acceptable technological level for rubber vulcanizates. The results gave indications that banana pseudostem modified fibres will find uses in rubber compounds.

Findings

This research work has characterized and established that banana pseudostem modify fibres will find uses in natural rubber compounds as a filler and a softener. Vulcanizates produced from such fibres can be used for materials in contact with petroleum fuels such as petrol, diesel and kerosene without any deleterious effect.

(a) Synthesis of biodegradable compounding rubber additives

Despite the natural occurrence of natural rubber, when used like other polymers, it is not easily biodegradable, that is, microbes cannot digest them and they do not rot away easily hence re-using, recycling or finding blends of polymer that are environmental friendly should be of paramount interest and importance, in order to save our environment. Below in Figures 23 to 25 are some pictures of the hazards already caused by non-biodegradable polymers

Fig. 23Polymers polluting our water sources and rivers

Fig. 24Polymers killing animals- birds

Fig. 25Polymers killing our sea animals

In other to solve these non-biodegradability problems, we have tried using locally available biodegradable materials as additives in NR compounding, Examples of materials already used are egg shell, different animal shells (snails, crabs etc), animal feathers and hairs (chicken, cow goat, pig), plant seed coats, plant fibres, etc. We have over 20 articles already published in this area, while work is still on-going. (Akinlabi *et. al.,* 2005 a, b, c d & e, Akinlabi *et. al.,* 2007 a, b, c d, e, f & g, Akinlabi *et. al.,* 2011 a, b &c,

Alayande *et. al.,* 2011, Alayande *et. al.,* 2014, Akinlabi *et. al.,* 2018, Oni *et. al.,*2020, Akinlabi *et. al.,* 2020)

Biodegradability of modified natural rubber blends with egg shell and watermelon rind - A particular research study aimed at determining the biodegradability properties of modified natural rubber vulcanizate compounded with eggshell shown in Figure 26 and watermelon rindshown in Figure 27 gave interesting results

Fig. 26 eggshell

Fig. 27 watermelon rind

The egg shells (ES) was obtained at an eatery along Alogi junction while Watermelon was obtained from traders at the Eleweran market, Abeokuta, Ogun State, Nigeria.Different steps for modification, compounding, curingof the vulcanizates and analysis were adopted.

Extracts from the results
The physico-mechanical properties of the vulcanizates with eggshell and watermelon rind showed comparable results with that of non-reinforcing standard filler, as well as the swelling and solubility results. Vulcanizates with Carbonized Eggshell (CES) and Carbonized Watermelon Rind (CWR) samples have superior mechanical properties when compared to the Uncarbonized Eggshell (UES) and Uncarbonized Watermelon Rind (UWR) and these enhancements were attributed to the higher carbon content of the CES and CWR. Compounding modified NR with the egg shell and watermelon rinds gave improved physico-mechanical

properties with a high solubility results in most solvents, giving an indication of a biodegradable material. The modified natural rubber vulcanizates with CES and CWR are more soluble than the NR-CB vulcanizate for all the solvent used, indicating that modification and substitution of CB with CES and CWR reduced its resistance to solvents. The order of swelling and solubility of vulcanizates by the solvents was butanol < hexane < CCl_4. The transport mechanism of solvents in the vulcanizates was also found to obey Fickan behaviour with high diffusion of solvents into the vulcanizates. The heat of sorption varies from exothermic to endothermic for the various vulcanizates in the solvent used. Summarily, modification of Natural Rubber allowed for better interaction with the eggshell and watermelon rind samples. Watermelon rind and Eggshell could be considered as a biodegradable additive in making some rubber products especially after carbonization and chemical treatments.

Biodegradability of modified natural rubber blends with acetylated cassava starch

Biodegradable plastic is an innovative means of solving the plastic disposal problem from the standpoint of development of new materials. Blending of polymeric materials has proved to be a successful method for preparing and developing material with properties superior to those of individual constituents. Hence, the blending technique becomes a desirable one and of commercial interest.

Biodegradation is considered a type of degradation involving biological activity. Biodegradation is expected to be the major mechanism of loss for most chemicals released into the environment.

Preparation of the blends

The blends were prepared by solution casting method. The natural rubber and modified natural rubber were separately dissolved in chloroform to form 5% (w/w) solutions. Cassava starch and acetylated starch (5% wt/wt) were gelatinized at 75˚C. Glycerol was added as plasticizer with 10% w/w relative to starch (dry basis). Thermoplastic starch/acetylated starch-natural rubber/modified natural rubber blends in the ratios of 100:0, 70:30, 50:50, 30:70 and 0:100 were prepared by mixing the solution together. The mixture was then homogenized by stirring for 10 minutes. The blends were casted onto glass petri dishes and oven dried at 50 °C overnight.

Mechanical test

Mechanical properties were measured in accordance with ASTM D-412 using an INSTRON tensile testing machine model 1781. The measurement was carried out on dumbell shaped specimens of 4 mm width and 40 mm length.

Determination of biodegradability of blends

The method of *Cheong et al., 2010*was used in the determination of biodegradability with modification. In situ environmental degradation was conducted on site on fertile garden soil (Morekete, Abeokuta). Initial weights of the samples were taken before they were buried at 10 cm below the soil surface. Samples were taken out of the soil monthly to measure their weights. This was taken for 12 months as shown in the results in Table 13. The weight loss was used as the parameter for determination of the

$$\text{Weight Loss \%} = \frac{(W_i - W_f)}{W_i} \times 100$$

Where W_i represents the initial weight and W_f represents the weight after each month

Table 13Biodegradability results over twelve months period

	Month/ %weight loss						
Sample	0	2	4	6	8	10	12
A	0	0	0	0	0	0	0
B	0	0	0	0	0	0	0
C	0	0	0	0	0	0	0
D	0	0	0	0	0	0	0
STS	0	41.43	86.3	100	100	100	100
ACS	0	37.66	79.4	100	100	100	100
NRS30	0	36.4	53.3	62.1	76.2	79.8	80.1
NRS50	0	26.5	40.3	41.6	43.7	48.7	52.9
NRAS30	0	35.5	52.8	61.6	69.7	71.8	74.3
NRAS50	0	25.42	37.15	42.31	46.53	51.36	52.62
ENRS30	0	38.2	57.8	64.6	68.7	71.3	73.8
ENRS50	0	34.52	44.42	47.97	48.98	52.03	53.04
ENRAS30	0	43.6	58.7	64.9	78.5	79.6	82.4
ENRAS50	0	34.23	43.86	47.35	49.63	53.18	54.32
NRS70	0	3.2	8.41	15.75	19.05	26.51	32.64
ENRS70	0	3.8	10.7	18.4	22.1	30.3	34.8
NRAS70	0	4.3	10.5	19.3	26.8	32.4	33.2
ENRAS70	0	6.7	14.61	18.16	22.47	32.96	37.27
DPLS30	0	34.7	45.7	48.2	52.4	62.4	74.1
DPLAS30	0	29.8	36.5	41.9	48.3	53.4	59.6
DPLS50	0	16.5	42.1	45.7	49.4	58.5	61.6
DPLAS50	0	47.8	56.5	56.5	56.5	57.07	59.8
DPLS70	0	1.7	13.7	17.6	20.9	24.7	30.8
DPLAS70	0	8.8	11.8	14.7	17.1	18.8	26.5
DPRS30	0	47.2	51.6	55.9	58.6	60.3	62.1
DPRAS30	0	45	63.8	65	67.5	69.3	71.8
DPRS50	0	24.5	39.4	41.3	43.6	47.4	48.2
DPRAS50	0	38.8	44.4	44.4	46.1	48.9	50
DPRS70	0	1.9	13.2	18.5	23.2	25.8	27.7
DPRAS70	0	3.2	16.4	22.6	24.8	25.2	26.5

A= Natural rubber, B= epoxidized natural rubber, C= deproteinized natural rubber, D= Depolimerized natural rubber, ENRAS30= 30%epoxidized natural rubber 70% acetylated starch, ENRAS50= 50% epoxidized natural rubber 50% acetylated starch, ENRAS70 = 70% epoxidized natural rubber 30% acetylated starch, DPRAS30= 30% deproteinized natural rubber 70% acetylated starch, DPRS = deproteinized natural rubber starch, DPLAS= depolymerized natural rubber acetylated starch, ENRS= epoxidized natural rubber starch

Findings

The result of this study showed that all the blends of modified natural rubber with starch and acetylated starch at different ratios of 30%, 50% and 70% in general, biodegraded in real soil environment. Biodegradability of the blends increased as the percentage of the starch and acetylated starch increased. The biodegradability was also enhanced due to the presence of modified NR in the blends most especially the blends that contained epoxidized natural rubber. The rate of biodegradability increases with time.

(d) Conversion of agricultural waste materials into useful rubber additives

Additives modify rubbers by improving the limitation aspects of rubber so as to have additional and novel uses that can serve as a substitute to the imported ones. Some additives in rubber have also been found to cheapen the cost of production of a given rubber article; to enhance a set of mechanical properties; to increase longevity of the article in service or to facilitate various shaping process that rubber may be subjected to during processing (Akinlabi, *et al.*2003a). Natural rubber on its own cannot perform the required engineering materials until some additives are added, the addition of other materials to natural rubber has led to compounding, which begins in 1839 when Charles Goodyear subjected a rubber compound to heat of a hot stove. Since that time, Charles Goodyear had proposed over series of items that could be made from hisnewly discovered, useful and versatile compound. My compounding experiences over the years has made me to conclude that the most important factors needed to be considered during compounding and before making a choice about the recipe to use are: (i) price of the compounding ingredients (i.e. are they economical as to reduce cost of the product?), (ii)processing methods (simple, available and affordable), (iii) equipment (easy availability, easy handling, operation and maintenance of the equipment) and(iv) properties of the anticipated product (i.e. will the chosen ingredients impart the expected properties and performance on the product?).

My team have successfully used over twenty (20)materials and their carbonized forms in rubber compounding. Some improved limitations of the rubber by acting as anti-degradants, fillers, plasticizers, softeners, tackifiers, colorants etc. and even as ancillary additives. We have over twenty (20)journal articles published in this area. (Okeimen and Akinlabi 2003, Akinlabi e*t. al.,* 2004a&b, Akinlabi e*t. al.,* 2005a,b&c, Akinlabi. e*t. al.,* 2006, Akinlabie*t. al.,* 2007a,b,c,d&g, Malomo e*t. al.,*2010, Akinlabie*t. al.,* 2011a,b&c, Akinlabi. e*t. al.,* 2013, Akinlabi e*t. al.,* 2014, Akinlabi e*t. al.,* 2015a&b, Akinlabi e*t. al.,* 2016, Akinlabie*t. al.,* 2017a&b, Akinlabi e*t. al.,* 2018a&b, Akinlabi e*t. al.,*2020 a & b)
Basically, a recipe for rubber to be compounded can contain combinations of some of the following ingredients:

(I) Vulcanizing agent: Vulcanization is a term generally used today to describe the reaction that produces the setting mechanism in rubbers. It affects the rate of vulcanization, cross-link structure, and final properties. Vulcanization increases the retractile force and reduces the amount of permanent deformation remaining after removal of the deforming force by the insertion of crosslinks between polymer chains forming a crosslinked molecular network. Commonly, sulphur is used as vulcanization agent. Sulphur which is suitable for vulcanization has to be at least 99.5% pure and it must not contain more than 0.5% ash. There are two types of sulphur which are cyclic sulphur (S_8) and amorphous sulphur. Other important vulcanization agents are selenium, tellurium, thiuram disulphides, peroxides, metallic oxides and quinone dioximes.

(ii) Accelerator: They are ingredient used in compounding to reduce the vulcanization time, by increasing the speed of vulcanization. Accelerators are divided into five classes according to their chemical structure application in the rubber industry. They are sulfonamides, dithicarbamates, thiazoles, guanidine, dithiocarbamates, and thiurams.

(iii) Activators: These additives are used to increase the vulcanization rate by activating the accelerator so that it performs more effectively. They are grouped into two namely the inorganic and organic accelerator activators. Examples of inorganic activators are zinc oxide, hydrated lime, litharge, red lead, white lead, magnesium oxide. Examples of organic activators are stearic, oleic, lauric, palmitic and myristic acids, and hydrogeneated oils from palm, castor, fish, and linseed oils.

(iv) Retarders: These ingredients are used to reduce the accelerator activity during processing and storage i.e. to prevent scorch during processing and prevulcanization during storage. The most common used retarder is cyclohexyl-N-thiophthalimide.

(v) Anti-degradant: These are ingredient used to retard the deterioration of rubber compound initiated by oxygen, ozone, heat, light, metal catalysis and mechanical flexing. In compounding, anti-degradant is required to give good aging properties to a compound and prolong its useful life.

(vi) Processing aid: These are the ingredient added to a rubber compound to facilitate processing operation, such as mixing, calendaring, extrusion and moulding. These materials react chemically to breakdown natural rubber and high Mooney viscosity synthetic elastomers chain and soften the rubber for easier processing or increasing the building tack after mixing. Examples of processing aid are fatty acids, metal salts of fatty acid and other fatty acid derivative, low molecular weight polymers and hydrocarbon oils.

Studies have shown that carbon black reduces the fatigue life of vulcanizate when use as filler in natural rubber compounding. Fatigue is said to be gradual weakening and eventual fracture of a material brought about by repeated cyclic deformation at strains lower than the breaking strain. My studies have been dedicated to utilize lingnocellulosic fillers such as coconut shell, wood, pineapple leaf, palm kernel shell, cocoa shell etc, and protein containing fillers such as chicken feather, goat hair, pig hair, etc, as fillers with aim of finding a replacement for the synthetic fillers through utilization of natural fillers. The use of agricultural waste material such as rice husk as filler have been documented and it was reported that such fillers minimize the cost, increase the productivity and enhance mechanical properties of product (Akinlabi, 2007). The following agricultural waste materials have been successfully used by my research team: *Pterocarpus Santaliniode Pericap, snail's shell and periwinkle shell, Jack beans (Canavalia ensiformis) and Sand Box Seeds (Hura crepitans), cowpea, cement klin dust, chitosan derivatives., egg shells, acacia seeds and moringa seeds, pig hairs, fowl feathers, cow hairs, pine cone, wallnut shell, watermellon rinds, termite mould soils, baobab seed, seed coat, pulp and fibre. Articles were published in journals on findings from the materials mentioned.*

(vii) Characterization of rubber seed oil and its potential uses

Oil was extracted from the rubber seed and characterized for its potential values. Characterization of the oil was mainly centred on: percentage yield, acid value, saponification value, iodine value, peroxide value andrefractive index. The results obtained indicated that the rubber seed oil can find uses in preparation of alkyd resin, binders, perfumes, mosquito repelling creams, pomades processing oils, etc

4.0 Constraints and advice on science and economic development

When there is low income, there will be poverty and hunger in the system. Estimates by the United Nations Food and Agriculture Organization (FAO) between 2014 – 2019 indicated that about 795 million people are hungry in a global population of about 7.6 billion people. Expectedly, 780 million people (98.1%) of the hungry people live in developing countries including Nigeria, a rather sad situation reflecting the deplorable condition of human wellbeing in these countries.The World Food Programme has also provided some disheartening statistics about famine worldwide, with about 805 million people not having adequate nutrition. The highest percentage of undernourished populations, regretfully, is also in Sub-Saharan Africa.Perhaps more worrisome is the manifestation of hunger in children, attested to in fetal growth restriction, stunting, wasting, and deficiencies of vital micronutrients. The consequence is that poverty has led to poor nutrition hence a way of reducing this is to add value to our raw materials by converting them into products that will attract cost and improve the living standards of the citizens. We have seen how our so called waste agricultural materials have been turned into useful polymer additives, this can be a source of income for those who ventured into it.

At one forum or the other, speakers and contributors have highlighted the multitude of problems plaguing science development and the technological capability of the Nigerian scientist to effectively harness the abundant natural resources for social economic development. The problems include shortage of high quality academic staff in higher institutions, brain drain, infrastructural decay, student related problems, such as overcrowded classrooms and hostels, poor quality students, increase in enrolment, inadequacy of laboratories, poor access to text books, inadequate ICT facilities, inadequate funding, poor investment in science, general absence of scientist in policy making positions at the national level, inadequate library facilities, poor networking and collaboration amongst scientists, apparent lack of University-Industry partnership, inappropriate research agenda and finally poor articulation of entrepreneurship development. The list is inexhaustible. The results of the afore-mentioned problems are numerous and complex. One of which is that there is a mismatch between Education and employment opportunities obviously deriving from a system which suffers from poor funding, structural deficiencies and academic imbalance. Secondly there is a yawning skill gap that cuts across the industries. Most dangerous of all these problems are graduates with poor communication skills, who lack analytical and problem solving skills, narrow knowledge base, inadequate professional carriage and poor professional ethics.

It is however heart-warming to note of recent that these problems are being addressed by the various Federal Government intervention programmes beginning with introduction of Entrepreneurship Education in the tertiary education curriculum, provision of Tertiary Education Trust Fund (TETFUND) research grant to Universities researchers and financial support to prospective Ph.D candidates that are lecturers. This is apart from the usual postgraduate scholarships and monies derived from Petroleum Development Trust Fund granted for foreign postgraduate studies.

Other problems that are seldom mentioned are the lack of political will of the three tier governments to rehabilitate scientific research institutions, generally low level of technical expertise for scientific research, lack of coordination of research results, lack of the capacity of laboratory technologist to arrest equipment failure and to service or maintain sophisticated instruments. Worst still is the

issue of private "wonder Schools" that litter the landscape, the so called "miracle center syndrome". The syndrome is so cancerous that if not checked, its impact on society might be worse than COVID-19 pandemic in the near future.
Some private, government schools and miracle centers have no science laboratories and do hire science apparatus and chemical from better but few established colleges to conduct WAEC/NECO Examination's practical. It is disgustful also to note that these schools churn out graduates with mostly A or B grade in science subjects. Who is therefore fooling who? Some, I am sorry to say this, some WAEC/NECO supervisors and ministry officials who are saddled with the responsibility of maintaining the rules and strictness in the conduct of such public Examinations don't also help matters. However I salute the courage of majority of school principals and proprietors, WAEC/NECO supervisors and Ministry officials and certain governments who had stood doggedly against this infectious educational malaise in our time. As a way of containing this trend, the Ministry of Education should continuously and conscientiously, monitor compliance and performance of the schools.

For all of the constraints enumerated above, there is no doubt that very well-articulated therapies have been suggested and made known to government. It will therefore take the political will and deliberate action of government to implement relevant sections of the annual budget to address them. Sadly this has not been the case as political considerations had always overridden the economic factor and the need to develop and advance science at our educational institutions in our geographical confine. But all of us gathered together today must realize that fostering the development of science education will necessarily serve as a spring board for acquiring research and technical capabilities for the provision of solutions to human challenges and environmental degradation. Remember that technology is the bedrock of civilization and industrialization.

A key issue in science development is science teachers. Fortunately science teachers are no longer scarce as they used to be about two decades ago, but how capable and skilled are these science teachers to arrest the intimidation of students by the sciences? Teachers are key elements in the implementation of all science programmes, hence teachers must be trained, retrained and encourage to improve themselves by enrolling in postgraduate programmes in core subject areas. Teachers must also be properly remunerated and granted incentives to encourage them. On a final note, the fundamental issue militating against science development is poor funding. We must streamline and encourage hard science skills at all levels if we hope to provide easier access to new knowledge.

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I wish to remind government that it is yet to implement the UNESCO standard and recommendation to the effect that all Nations of the world must expend 26% of its GDP on Education annually. This is what ASUU has been saying over years. The European Union on the other hand has made vocational and technical training a key factor in economic policy and the fight against unemployment. So what has happened to our Technical Colleges? There is urgent need therefore, for government at all levels to have a clear non-negotiable policy and direct investment in the education sector, since the wealth or poverty of Nations depend on the quality of her higher Education.

5.0 Conclusion

Mr. Vice Chancellor Sir, I have in the course of this lecture attempted to highlight some of my research activities in order to buttress the importance of Chemistry as a discipline on a road march to discoveries, innovations, life sustenance and economic development. My doggedness and perseverance has however seen me through to some accomplishment. I have attempted to link the underdeveloped status of Nigeria to low capacity natural resource utilization. The wealth of a nation in terms of natural resource endowment does not automatically translate into wealth and well-being of its citizen. That nation must transform these resources into wealth for the benefit of the people. This is the challenge we are facing now and any developmental programme based on local resource utilization must begin from where we are.

Over the years, I have successfully contributed to knowledge in many areas but more precisely in the field of polymer and specifically on natural rubber. *If all the mentioned modifications and substitutions of the additives used in polymers are adopted by our technologists, there will be increase in the economic growth and those who engage in it will have higher earnings. The problem of change is what our technoligsts are afraid of.* I have designed and introduced the method of natural rubber depolymerization and synthesis of low molecular weight rubber, which are gaining wider interests and acceptance among researchers in the field of polymer. I have published seventy five (75) articles in the field of natural rubber. I designed method of carrying out Mooney relaxation of rubbery materials, developed a simple relationship between the least relaxation point of rubber and the plasticity of the rubber, designed method for the production of epoxidized low molecular weight natural rubber and thioglycollic acid modified epoxidized low molecular weight natural rubber, designed methods for determining the level of filler comparability in modified low molecular weight rubber matrix, designed methods for carbonizing agricultural waste materials for use as additives in polymer processing, developed simple stress-strain tests for determining the crosslinking properties of vulcanizates, modifying natural rubber latex and compounding the latex with local additives for new applications. All these designs, procedures and methods have generated new set of information and data in polymer industry, while two have been patented and they are gaining wider acceptances and been referenced by scientists who have ventured into polymer modification, characterization and processing.

I have also attempted to share with us aspect of our work on the application of Chemistry in the health sector, acquiring wealth and in the development of industrially useful value-added products from locally available renewable resource materials that would create wealth, employment and raise the quality of living standards of the people. In our present situation, the world is now global and information are no more hidden, hence we need to be honest and earnest about our facts and figures for us to be key players in worldwide global research affairs. Our research should also impart positively on our society leading to discoveries and innovations on our immediate needs. We adopt the adage, necessity is the mother of invention.

Mr. Vice-Chancellor sir, twenty nine years (1992 to 2021) down the road, I am proud to tell my friend, who challenged me then, on "what will I do with chemistry", and to tell him now that I have used my knowledge of Chemistry to provide for the needs of my people. I do not know how much success I have made, but I know that I am more self-confident discussing my works on polymer modifications and applications, local sourcing of renewable resource materials, biopolymer applications, biodegradable and multifunctional polymer additives, etc. I dare to say that I have more than half-way met the challenge.

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Advice to all Researchers

Although research grants are not easily come by but researchers should stop procrastinating and continue to make noise on their findings maybe one day it will come out of the laboratory and get commercialized.

What is in this life that is not Chemistry?

AIR - CHEMISTRY
FOOD - CHEMISTRY
WATER - CHEMISTRY
GASES - CHEMISTRY
ENVIRONMENT - CHEMISTRY
NATURAL RUBBER - CHEMISTRY

Adding Value to Natural Rubber sustains Life and Economic Development.

Thank you for listening

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