



**FEDERAL UNIVERSITY OF TECHNOLOGY
MINNA**

**PROCESS ENGINEERING:
A NEGLECTED TOOL IN WASTE
MANAGEMENT AND PRODUCT
UPGRADE IN NIGERIA**

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*B.Sc. (Romania), PGD.Ed (UNN), M.Eng. (ESUT),
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1.0 INTRODUCTION

This presentation shall be in 3 parts – industrial liquid effluent treatment and air pollution abatement, upgrade of a natural product and agricultural solid waste conversion.

1.1 Brief History of Chemical Engineering/Process Engineering

Chemical engineering began as a distinct profession in England towards the end of the 19th century. The chemical engineer emerged gradually from the chemist who has always been a partner of the engineer. Towards the end of the 19th century, there was a dire need for the training of chemical engineers. Consequently, a meeting, the first of its kind, was held in England in 1879 by a group of people who were interested in the commencement of the training of chemical engineers. But only America seized the initiative in the development of this branch of engineering. The first curriculum ever to be designed for the training of chemical engineers was established at the Massachusetts Institute of Technology (MIT) in 1888. By comparison, the Nigerian Society of Chemical Engineers (NSChE) was established in 1970 and formerly incorporated in 1980 (Okafor, 1999). Up to now however, good chemical engineering practice is still founded on the sound application of two aspects of empiricism and theory (Aneke, 1989).

1.2 Field of Chemical/ Process Engineering

It is called Chemical Engineering in U.S.A. and Process Engineering in Britain. The areas of specialization include Reaction Kinetics, Reactor Design, Thermodynamics, Fluid and Particle Mechanics, Transport and Storage of Fluids, Handling of Bulk Solids and Packaging of Solids and Liquids, Size Reduction and Size Enlargement, Energy Utilization, Conversion and Resources Conservation, Heat and Mass Transfer Operations, Cryogenic Processes, Process Control and Instrumentation,

Process Machinery Drives, Process Economics, Waste Management, Biochemical Engineering, Waste Recycling and Beneficiation of Raw Materials .e.t.c

1.3 Need for Safety and Hazard Studies

Safety and environmental impacts of chemical substance were raised even from the onset. For example silent spring, published in 1962, which alerted the general public to the harmful effects of Dichoro-Diphenyl-Trichloroethane (DDT), the 1974 Flixborough disaster in the United Kingdom and more recently the 1984 disaster (Phosgene Leak) in Bhopal India where more than 4,000 people died.

1.4. Water Pollution and Control

The world environment is threatened by incessant pollution with untreated industrial wastes. In order to address this problem, World Commission on Environment and Development adopted Sustainable Development Strategy as a policy at the conference on the Human Environment, held in Stockholm, 1972. Some sources of these wastes are effluents from dry-cleaning services, bottling company effluents, some domestic effluents, chemical and allied industrial effluent e.t.c. The approach to solving these environmental problems varies from place to place. The European Union enforces the Integrated Pollution Prevention and Control (IPPC) and landfill directives (Aneke and Okafor, 2008) as regional strategy while, Nigerian approach avoids the expensive as well as capital intensive environmental control measures. According to Iyang (1978), environmental pollution is accentuated by unprecedented population and urbanization growth rate.

Discharge of coloured industrial/ domestic effluents imparts to the receiving waters colour which has the ability to inhibit growth of the desirable aquatic biota necessary for self-purification (re-oxygenation). The colour compounds also have a

tendency to chelate metal ions (formation of ring structure with metal ions) and thus become directly toxic to aquatic biota (E.P.AR2-73-167,1973).

2.1 ACTIVATED CARBON

Activated carbons are industrial carbons used in domestic as well as in chemical industries and consumer products to electronic industries (Dai, 1994; Byme and Nagle, 1987 and Ryu, 1994). This large potential use comes from the fact that activated carbon with good internal surface area, porosity and adsorption characteristics can be manufactured by choosing the right kind of raw material and controlling the process condition. However, feed stock of agricultural origin such as wood, coconut shells, agricultural wastes or coalified plant matter such as coal, lignites etc. are the major raw materials for commercial activated carbon (Roy, 2002).

2.2 Policy on Environment

The importance of water among living things and as a universal solvent cannot be over-emphasized. Therefore, World Health Organization (WHO) has recommended a standard quality on water (Abdulsalam and Ajiboso, 2003). Adsorption process is therefore an efficient means for the removal of contaminants in industrial wastewater. One of the most useful attributes of activated carbon is it's ability to be economically regenerated and reused, even after the spent materials has become fully saturated with adsorbate.

2.2.1 Activation

Two methods of activation are, (i). Thermal activation and (ii) Chemical activation. Whether thermally or chemically activated, the excellent adsorption properties of activated carbon stem from a large surface area of upto $1500\text{m}^2/\text{g}$. (Report No. 923/1/00).

2.2.2 Thermally Activated Carbon (TAC)

The AC precursor is usually processed in a carbonised form and activation normally takes place at temperature of 800-1100°C in the presence of steam or CO₂, N₂ etc. Carbon activated in this way generally exhibits a fine pore structure, ideal for adsorption of compounds, in both liquid and vapour phase applications (Ben,2000). During the process of activation, the spaces between the elementary crystallites become cleared of various non-organised carbon and impurities. Since the average micropore area increases due to heat treatment the adsorption capacity of carbon also increases.

The influence of activation temperature on micro and mesoporosity of synthetic activated carbons was equally studied by Warner (2003) and from the results of study, it was confirmed that elevated activation temperature actually leads to the formation of microstructures without significant change in mesostructures.

2.2.3 Chemically Activated Carbon (CAC)

In chemical activation of carbonized precursor, the raw material is impregnated with strong dehydrating agent, typically phosphoric acid, zinc chloride, potassium hydroxide etc. The carbonized carbon is mixed with the chemical activating agent into a paste and then heated to temperatures of 500-800°C. Chemically activated carbons generally exhibit a very open pore structure ideal for the adsorption of large molecules (Ben, 2000) In another study, temperatures of 700, 800 and 900°C were used in chemical activation for nitrogen adsorption, the adsorption capacity and pore size distribution were highest at 900°C. (Hu and Vansant, 1995). The equilibrium relationship between adsorbate concentration at a fixed temperature is termed adsorption isotherm.

2.3. TYPES OF ADSORPTION

2.3.1 Chemical Adsorption (Or Chemisorption)

This involves forces much stronger than in physical adsorption. This is the result of chemical interaction between the solid (adsorbent) and the adsorbed substance (adsorbate) (Steve and Erika, 2002). Chemisorption occurs when the bonds to the surface result from an exchange or a sharing of electrons. The adsorbed molecules are held to the surface by valency forces of the same type as those occurring between atoms in molecules. Large heats of adsorption are the values of the same magnitude as the heat of chemical reaction, 5-100kcal/gmol (Bratu,1970). (Suzuki, 1990 and Steve and Erika, 2002).

2.3.2 Physical Adsorption

In physical adsorption, the forces attracting the fluid molecules to the adsorbent surface are relatively weak (Van der Waal or London forces), and the heat evolved during the exothermic adsorption process is of the same order of magnitude as the heat of condensation, 0.5 to 5kcal/gmol. Equilibrium between the solid surface and the gas molecules is usually rapidly attained and easily reversible, because the energy requirements are small (Suzuki, 1990).

2.4 REGENERATION OF ACTIVATED CARBON

Activated carbon filters have a limited lifetime. The surface of the AC will eventually be saturated with adsorbed pollutants, and no further purification will occur. This is called breakthrough ie the pollutants have broken through the filter to emerge in the treated water (MSU) Extension Water Quality Bulletins, 1997). Knowing when the breakthrough will occur and thus when to replace the cartridge is a major problem with AC treatment. Therefore, the AC needs regeneration.

The breakthrough concentration is determined by the process specifications, or government regulation. At breakthrough

concentration, adsorption stops, then the activated carbon bed has needs for regeneration. In the past applications, once the activated carbon has reached equilibrium with the contaminant or become “Saturated”, the contaminated carbon was sent to an off-site regeneration facility or the carbon was disposed of, usually in a landfill (Jon and Tuttle, 1997).

2.5 KINETICS OF ADSORPTION

Generally adsorption processes proceed through various mechanisms: Convective/ diffusion mass transfer within the surrounding fluid, External mass transfer of solute onto sorbent, Intraparticle diffusion. Venkata and Karithikaya (1997) in a study on removal of lignin and tannin colour from aqueous solution by adsorption onto activated charcoal showed that the adsorption process was of a first- order kinetics represented by.

$$\log \frac{C}{C_e} = k \frac{t}{2.303} \quad (2.1)$$

The most commonly used equilibrium models to understand and predict the adsorption systems are Freundlich, Langmuir and BET equations. These are given in equation 2.2, 2.3,2.4, 2.5 & 2.8 respectively.

$$\frac{C_b}{C_o} = K_t \quad (2.2)$$

Cooney; (1999) and (Mckay 1996),

$$\text{Log} \frac{C_b}{C_o} = \frac{N_o}{D} K. \epsilon. t_b. V. 1000A \quad (2.3)$$

2.5.1 Freundlich Equation

Herbert Max Finley Freundlich, a German Physical Chemist proposed an empirical equation as

$$q = K_f C^n \quad (2.4)$$

which on taking the logs and rearranging gives

$$\log q = \log K_f + n \log C \quad (2.5)$$

The coefficient K_f and n can be estimated from the slope and intercept of a graph of $\log q$ versus $\log C$ (Venkata and Karithikaya, 1997). This model is often a better fit for physical adsorption systems, particularly for adsorption from liquid (Warren et al, 1993 and Perry et al, 1997), where the actual identity of the solute is not known as in the adsorption of coloured substances from effluents (Treyball, 1981). This model is used to determine the adsorptive capacity of activated carbon over a range of different concentrations. The adsorptive capacity of activated carbon will increase as the concentration increases until the maximum saturation capacity is reached. From eqn (2.5), n is also known as heterogeneity factor and K_f the adsorption constant. The Freundlich model assumes that the uptake of any adsorbate occurs on a heterogeneous surface by multilayer adsorption and the amount of adsorbate increases infinitely with increase in concentration and when n is greater than 1.0, the conditions are favourable for adsorption (Egwim and Okafor, 2011).

2.5.2 Langmuir Equation

Here the rate of attachment to the surface is proportional to the fraction of surface area covered. The driving force is the concentration in the fluid, and the area is the amount of the same surface.

At equilibrium, the rates become equal

$$K_1 C (1 - \theta) = K_2 \theta \quad (2.6)$$

The useful form of equation (2.6) is,

$$q = \frac{q_m k_a C}{1 + k_q C} \quad (2.7)$$

Taking reciprocal and rearranging of equ (2.7) gives

$$\frac{1}{q} = \frac{1}{q_m} + \frac{1}{k_q q_m C} \quad (2.8)$$

Equ (2.8) could be expressed in a standard format as given below;

$$\frac{C}{q_e} = \frac{1}{Q_b^0} + \frac{C}{Q^0}$$

From equs (2.3) and (2.5); (2.9)

where C is the initial concentration of colour (mg/litre⁻¹), C_e is the equilibrium concentration of colour (mg/litre⁻¹), t is time (min), and k is the rate constant (min⁻¹). C_o=Initial concentration of pollutant (mg/l); ε= Porosity of the bed; t_b= Time until breakthrough (min) K= Quasi chemical rate constant from Bohart and Adams theory (l/mg-min); V = Volume of Fluid

C_b – breakthrough concentration of pollutants (mg/l⁻¹)

C_o – Initial velocity or loading rate (m/min); N_o – capacity of the media for each pollutant in multi-compound solution (mgm⁻³ of filter volume, 1000 – the conversion factor between litres and cubic metres, A- a constant (min)

q = weight adsorbed per unit weight of adsorbent (mg); C = concentration of fluid (mg/litre⁻¹); K_f is a coefficient/constant (dimensionless), n ≅ 0.5.

where q_m = q for completely monolayer (g adsorbate/g solid); k_a = a coefficient (kg/m³ or g/cm³); and C is the equilibrium concentration of sorbate (mg/litre⁻¹) or ppm or g/cm³ or mol/m³

Q^o and b are the Langmuir constants related to capacity (mgg⁻¹) and energy respectively.

2.6 DESIGN OF A STEAM CHEST

Steam chest is the box or chamber from which steam is distributed to the cylinder of a steam engine, steam pump etc and usually contains one or more valves, called valve chest or valve box (Henry, 2003). Here, the steam chest is used to regenerate activated carbon when it becomes saturated.

2.7. SHEA BUTTER BENEFICATION

Shea tree (also called *Vitellaria Paradoxa* or simply *V. Paradoxa*) in West Africa, is predominantly found in countries like Burkine Faso, Mali, Nigeria, etc. It takes a minimum of 28 – 30 years to mature and bear fruits (Walter et al., 2003). Unfortunately, only a few of these countries know the economic benefits of the plant if properly harnessed. More often than not the locals harvest the wood for domestic energy supply in cooking and in heating homes during harmattan. Only an insignificant few use obsolete traditional methods to process shea nut to shea butter with consequent low yield, loss of raw materials and of low quality grade (E). This grade attracts poor pricing and at times out-right rejection of the product at the international shea market. Unknowingly to these communities, the shea tree is capable of liberating them from the shackles of poverty. It is estimated that the quantity of shea butter produced from these countries is put at 600,000 tons with only 350,000 tons exported in 2008. This low volume of export is not unconnected with the poor standard of the butter produced from this region.

Lovett (2004) specified that 50 – 60% of shea tree in West Africa are domiciled in North Central States of Nigeria; Niger, Kwara, Nasarawa, Plateau, Kaduna and some parts of Kebbi, Bauchi Kogi, F.C.T and Oyo (Munir et al., 2017). Meanwhile a country with very little or no shea tree (United States of American) has established what is known now as American Shea Butter Institute (ASBI) for Education and Training, Analytical Testing and Grading Services, Post Extraction Management Certification International Shea Butter Convention (ISBC). All these efforts

are designed to expose countries with Shea trees of the wide spectrum of opportunities in the Shea industry, spanning from local business in Subsaharan Africa to various global markets.

The demand for Shea butter produced in West Africa has increased by over 1200% in the last ten years. In 2008, an estimated 350,000 metric tons of Shea kernels were exported from Africa with a market value of approximately USD120 million. The demand for this valuable product continues to grow and for the past 10 years, demand for shea butter products has grown in both the European Union and the United States of America (Saba et al., 2017). The net worth for this demand is about USD 30 billion by the year 2020.

This again underscores the need in Nigeria to key into this global demand-supply for shea butter by producing good and consistent shea butter quality.

As a result of poor and uncontrolled processing methods, shea kernels generally undergo hydrolytic and oxidative degradation, are effected by aflatoxin and other harmful micro-organisms during the post-harvest processing and storage (Saba et al., 2019). This defective and obsolete approach in shea butter production has been overcome by the use of more scientific methods for grades A or B (Saba et al., 2018)

The traditional approach even though has a lot of defects but the butter obtained from this method is better than solvent extraction and mechanical expression which is highly refined and hence contain little or no bioactive components while the shea butter obtained from mechanical expression involves the use of expellers or hydraulic press which exerts high pressure on the heated kernels to express the shea oil. The oil obtained from mechanical expression has very high FFA and requires refining before use (Julius et al., 2013). On the other hand, the traditional method of shea butter extraction is still the most preferred (Loveth, 2013) because of its retention of the bioactive fractions.

In an earlier study to investigate the effect of moisture content using traditional method, Saba et al., (2018) asserted that moisture in agricultural materials affects the physical and mechanical properties, storability, handling and processing of biomaterials and by extension shea nut and shea butter. The study revealed that shea nut dried with a locally fabricated dryer gave higher yield than the one dried under uncontrolled atmospheric temperature conditions. Also other quality indicators, for example IV, FFA etc. were significantly improved and the shea butter obtained approached the international standard grades A and B.

2.8 AGRICULTURAL WASTE IN ENHANCING RAW RUBBER QUALITY

Rubber material is critical to modern industrial civilization with applications in all critical sectors of human endeavours. To an engineer, rubber is considered to be a polymeric material which has the ability to stretch to at least 100 % of its initial length without permanent deformation, and subsequently returned to its original configuration after being stretched (Phomrak and Muenduen, 2017).

Rubber is used in modern transportation in the making of tyres, in sealing fluids (O-rings), conducting fluids (tubes and hoses), storing energy (bungee cords), transmitting energy (drive and conveyor belts), absorbing energy and providing structural supports (engine seats, vibration dampers and bridge bearings). Raw rubber in its raw and uncured state possesses low strength, highly dimensionally unstable under thermal and loading conditions and practically has no use (Hamed, 2007, Visakh *et al.*, 2012). It is required to be vulcanized and reinforced in order to perform satisfactorily under thermal and loading (quasi-static and dynamic) environments. After vulcanization of natural rubber the initially soft-weak and dimensionally unstable rubber

becomes transformed into a strong elastic material with high and reversible deformation and better mechanical properties. Nevertheless, the inherent strength of this vulcanized rubber can further be improved upon by incorporating reinforcing filler (stiff particulate or fibrous materials) into the rubber matrix to obtain composite with improved tensile strength, modulus and durability required for structural and engineering applications (Visakh *et al.*, 2012).

Reinforcement in the context of polymer composites simply implies increase in the load bearing capacity or in the mechanical properties such as tensile strength and modulus of the composite (Kannan and Ahmad, 2013).

The rubber industries rely on carbon black and silica as their main sources of rubber reinforcement. The use of silica filler is expensive while carbon black (acetylene black, charcoal black, and furnace black, lamp black and thermal black) is produced through incomplete combustion of heavy petroleum fractions and these are exhaustible and results to negative environmental health impacts (cancer) (Verma *et al.*, 2013, Shah *et al.*, 2016). Green manufacturing processes is an attractive option and form part of the motivation to create attention towards renewable and readily available eco-friendly materials such as cellulose that can possibly be modified to meet the requirements of a reinforcing phase in polymer (rubber) composites (Gilberto *et al.*, 2011, Eldho *et al.*, 2013, Lee *et al.*, 2014).

Cellulosic fibres can be extracted from coconut husk, bamboo and cotton linters through alkaline-peroxide treatment. These agro wastes are cheap and contain relatively high holocellulose (cellulose and hemicelluloses) contents and their huge abundance in Nigeria, renewability, non-toxicity, bio degradability and eco- friendliness make them attractive (Rowell, 2008, Ogunwusi and Onwualu, 2011, Serge and Fernand, 2013, Kalia *et al.*, 2011).

Natural fibre have limitations such as moisture absorption, quality variations, low thermal stability, and poor compatibility with the hydrophobic polymer matrix (Chandrasaha *et al.*, 2014). These limitations can be eliminated or minimised by surface modification through delignification. Delignification is the removal of the structural polymer lignin from the plant tissue. The attraction for use of cellulosic nanoparticles in polymeric composite materials is the large active surface area they offer in the enhancement of surface interaction and flexibility between the non-polar hydrophobic polymer matrix and the highly polar hydrophilic cellulose phase (Saheb and Jog, 1999). In addition, at nanoscale materials behaviour will change dramatically from what is obtainable at macro- or micro- scale due to their large active surface area and quantum effect (Farzana *et al.*, 2006).

3.0 RESULTS AND CONTRIBUTION TO KNOWLEDGE

3.1 Adsorbent Characterization and testing.

- I. The adsorbents after preparation were characterised and then used to treat coco-cola waste waters. There was a significant reduction in concentration of pollutant indicators, the surface area, fixed carbon, pore volume etc. were found to be higher than some commercial adsorbents. The waste water from the coco-cola plant was highly polluted but these pollutants were drastically reduced after it was treated with the adsorbents (Okafor and Aneke, 2006). The process is mono-layer and therefore physical in nature. See Tables 3.1 and 3.2
- ii. On using different agents on coconut shells to prepare other adsorbents, it was found that samples treated with KOH have least percentage ash content when compared with commercially available activated carbon, the bulk density increased with increases in activation temperature according to Dim and Okafor (2012). The lower the ash content, the

higher the amount of carbon derived. Chemical activation agent, carbonization temperature play significant roles in the development of activated carbon.

- iii. In yet another work by Okafor and Dim, (2013), it was shown that there is a strong relationship between fixed carbon and capacity. A very high amount of fixed carbon was found to be 93.9% when H_2SO_4 was used as chemical activation agent in the preparation of adsorbents using palm kernel shells for industrial effluent purification, the ash content was only 2%.
- iv. The methylene blue number approach was also used to elucidate capacity for some of the developed adsorbents: bone, wood and coconut shells. On treatment of the developed adsorbents with methylene blue, there was a significant reduction in absorbance from 0.790 to 0.105 using European methodology and 0.998 to 0.175 (American methods) when the adsorbents were activated chemically. Here again, thermally activated wood using European procedure gave the best reduction in absorbance (0.790 to 0.059) as against 0.998 to 0.083 obtained using American method. A two-way ANOVA showed that there is a significant difference in the average time and no significant difference in the average effects of the variables (for thermally activated adsorbents using both methods) at 0.05 level of significance. But for chemically treated adsorbents using American method, time and average effects of the variables is highly significant at 0.05 level of significance while the European approach showed a significant difference at all levels of significance (Okafor and Aneka, 2007). Therefore the European method could be adjudged the better when a mixture of wood, bone and coconut shell treated chemically was used for the process.
- v. Cow bone and wood were also used as the precursor for adsorbent development. The samples were treated chemically and thermally. The results confirmed a monolayer coverage using both bone and wood. Chemically developed

bone and wood were found more suitable in the purification of dry-cleaning and coca-cola effluents. This absorption process is monolayer type with the Langmuir constants very adequate to justify their use in the purification of coca-cola and dry-cleaning effluents. (Okafor, 2012).

3.2 PHYSICAL CHARACTERISTICS OF DEVELOPED ADSORBENTS

Four adsorbents were developed A,B,C and D. According to Okafor and Aneke (2006) they were tested on coca-cola effluent.

| Sample | Surface area (cm ³ /kg) | Bulk density(g/cm ³) | Ash content(g) | Fixed carbon(g) | Pore volume | Porosity ϵ P | pH |
|--------|------------------------------------|----------------------------------|----------------|-----------------|-------------|-----------------------|-----|
| A | 1.053 $\times 10^{-2}$ | 6.52 | 0.10 | 0.90 | 0.82 | 1.76 $\times 10^{-1}$ | 7.0 |
| B | 1.372 $\times 10^{-2}$ | 5.89 | 0.089 | 0.92 | 0.90 | 1.56 $\times 10^{-1}$ | 6.0 |
| C | 2.152 $\times 10^{-2}$ | 11.36 | 0.19 | 0.81 | 0.65 | 2.14 $\times 10^{-1}$ | 5.0 |
| D | 1.729 $\times 10^{-2}$ | 10.12 | 0.16 | 0.84 | 0.35 | 1.24 $\times 10^{-1}$ | 6.0 |

Table 3.1 Physical properties of the adsorbents
Source; Okafor and Aneke, 2006

The reduction in the amount of pollutants contained in the effluent after treatment with the developed adsorbents are shown on Table 3.2 with their values before treatment and WHO highest desirable level.

Table 3.2 Physical and biochemical properties of coca-cola effluent before and after treatment

| | Bone Based Adsorbent | | | Wood based adsorbent | Bone based Adsorbent | Effluent value | WHO highest desirable level (1997) | Maximum permissible level |
|--------------------------------|----------------------|-------------------|-------------------|----------------------|----------------------|-------------------|------------------------------------|---------------------------|
| | A | B | C | | | | | |
| Ph | 6.50 | 7.00 | 6.50 | D | 7.00 | 4.15 | 7.0-8.5 | 6.5-9.2 |
| Turbidity | 20.30 | 9.52 | 12.47 | | 2.59 | 395.00 | 0.4 | 4.00 |
| Colour(Hazen) | 5.00 | 2.00 | 5.00 | | 2.00 | 70.00 | 5.00 | 50.00 |
| ----- | 300.00 | 214.00 | 290.00 | | 189.00 | 385.00 | 750.50 | 750.50 |
| Total dissolved solids(ppm) | 199.80 | 142.52 | 193.4 | | 123.88 | 256.41 | 500.00 | 500.00 |
| Temperature (°C) | 29.50 | 28.00 | 30.50 | | 27.00 | 30.50 | 21 | 39.00 |
| Refractive index | 1.33 | 1.33 | 1.33 | | 1.33 | 1.3345 | - | - |
| Density (Kg/m ³) | 1000.00 | 1000.00 | 1000.00 | | 1000.00 | 1006.00 | - | - |
| Total suspended solids(mg/l) | 0.04 | 0.025 | 0.035 | | 0.02 | 0.06 | - | - |
| Total Solids(mg/l) | 0.03 | 0.02 | 0.02 | | 0.01 | 0.04 | - | - |
| DO(mg/l) | 6.00 | 3.00 | 5.00 | | 2.00 | 7.00 | - | - |
| BOD ₅ (mg/l) | 36.00 | 21.00 | 127.00 | | 14.00 | 45.00 | 100.00 | 100.00 |
| Chromium (mg/l) | Nil | Nil | Nil | | Nil | Nil | 0.05 | 0.05 |
| Sulphate(mg/l) | 32.00 | 17.00 | 20.00 | | 12.00 | 56.00 | - | - |
| Chloride (mg/l) | 80.00 | 40.00 | 60.00 | | 20.00 | 160.00 | - | - |
| Nitrate (mg/l) | 0.00 | 0.00 | 0.00 | | 0.00 | 0.1 | - | - |
| Total Microbial Count(cell/ml) | 5×10 ⁶ | 3×10 ⁶ | 4×10 ⁶ | | 2×10 ⁶ | 6×10 ⁶ | - | - |
| Iron(mg/l) | 1.00 | 0.00 | 1.00 | | 0.00 | 20.00 | 1.00 | 1.00 |
| Odour | Partially removed | Totally removed | Objectionable | | Odourless | offensive | odourless | |

(Source: Okafor and Aneke, 2006)

3.2.1 EFFECT OF SOME IMPORTANT PROCESS VARIABLES.

- i. The effects of pH and temperature. It was observed that the process was exothermic in nature and also highly dependent on the pH values. Under different temperature conditions, 96.6% reduction in concentration of pollutants was achieved while 143.10% was obtained at various pH values. Wood is therefore recommended for his liquid phase adsorption process (Okafor and Aneke, 2007).
- ii. On using chemically treated wood and bone to purify dry - cleaning effluent at a low pH, over 100% purification was achieved for the two cases in 5 minutes. This again confirms the fact that adsorption processes are highly dependent on pH (Okafor, 2011). The extent of purification decreased markedly as the pH of the sample solution was increased.
- iii. Padamus Candelabrum stem, a new precursor was used to prepare activated carbon using NaOH, ZnCl₂ and H₃PO₄ as chemical activation agents. Surface morphology, elemental composition and functional groups were analyzed with SEM, energy dispersive EDX, XRD and FTIR spectroscopy respectively. The image analyses showed the presence of both micropores and mesopores in adsorbents. The H₃PO₄ activated carbon had the maximum surface area (2648m²/g), pore volume (1.683 cm³/g) and highest adsorption for iodine and methylene blue and were 541 and 105 mg/g respectively. This precursor is abundant, easy to access, inexpensive and readily available according to our findings (Dim et al., 2020).

3.2.2 ADSORPTION OF HEAVY METALS

- I. In a study by our research team, Dialium Indum and Santalum Album Fruit shells were used to develop adsorbents for the removal of cadmium ions from aqueous solution. The fact that FTIR Spectra contain -OH, COO- functional groups, should be responsible for binding of metal

ions on the developed adsorbents were revealed by the good fit of data into Freundlich Isotherm indicating multilayer adsorption on the adsorbents, The surface areas are extraordinarily high, extent of removal of pollutant depends on Cd ion concentration, contact time, adsorbent dosage and pH, (Jibrin et al., 2016)

- ii. Santalum Album (Sandal fruit shells) activated carbon was again used by our team to study C_u^{2+} and Pb^{2+} ion uptake from an aqueous solution. The equilibrium sorption conformed better to Freundlich model than the Langmuir isotherm model as shown with high correlation coefficients (>0.95). The biosorbent is an excellent biosorbent which can be utilized as an inexpensive biosorbent for the removal of heavy metals from an aqueous solution. The kinetics of C_u^{2+} and Pb^{2+} were modeled using the pseudo-first and pseudo-second order kinetic equations and the results showed that pseudo-second order model provides better correlation with adsorption data on C_u^{2+} and Pb^{2+} and these correlation values are more than 0.9 (Akoji et al., 2015)
- iii. In another study by us using Ahoko Kaolinite clay, the activated clay is good for the uptake of both Cu (ii) and Cr(vi) ions from waste water if the clay is chemically activated. The kinetic study reveals that pseudo-second order model shows a good fit in case of all reactions and initial concentrations. The thermodynamic parameters were evaluated and were found to be positive, negative and positive for ΔH , ΔG and ΔS respectively indicating an endothermic process, spontaneity and feasibility of process and a high degree of randomness respectively. It was observed that Ahoko clay has a higher affinity for adsorption of Cu(ii) ions than those of (Cr(vi) ions. (Dim, et al., 2020). This is a good example of selective adsorption and also an important contribution.
- iv. Again our team used batch process to conduct adsorption study to remove Cr(vi) and Fe(iii) with HCl acid modified

(HMC) and acetic acid modified Umunze clay (AMC). Activated clay morphology, chemical properties were studied by SEM, XRF, cation exchange capacity (CEC), XRD and BET. The effects of process variables were also investigated. Acid activation increased the BET surface area from 84.223 m²/g to 389.37m²/g (HMC) and 319.955m²/g (AMC), total pore volume to 0.2168 and 0.2285cm³/g respectively. XRF, SEM and XRD measurements show disintegration and porous structures of the acid treated clays. HMC has a maximum removal of Cr(vi) and Fe(iii) of 79% and 90% respectively while equilibrium was attained at 50 and 90 min respectively. ΔH is positive which indicates an endothermic process and this suggests the interaction of metal ions with acid modified clays. ΔS is also positive and suggests a strong affinity of ions for adsorbent and a high degree of randomness. However, ΔG is negative which implies that the process is spontaneous. The more negative the ΔG is, the more favourable and energetically spontaneous the reaction is (Dim et al., 2021).

3.2.3 MECHANISM AND THERMODYNAMICS OF HEAVY METAL ADSORPTION

- I. Mechanism and thermodynamics: In our study (Jibrin et al., (2015), the sorption mechanism for the modified form of the velvet tamarind is linked to the role played by the essential stretching of the functional groups like (-OH). H₂SO₄ decreased low molecular weight lignin compounds thereby increasing the concentration of (-OH) and (-COOH) groups. Ashing also increased the concentration of Si-O-Si and Si-OH which have the functions of heavy metal removal. The high correlation coefficient (R^2) of 0.79 and 0.80 shows that velvet tamarind to be an excellent biosorbent. Negative value of G shows that the process of adsorption is spontaneous for C_u^{2+} while that of Pb^{2+} is non spontaneous, Adsorption of C_u^{++} is

endothermic but that of lead is exothermic. The biosorbent is eco-friendly and cost-effective. In other words ΔG for Pb^{2+} is positive which helps to make the process selective if there is a need to work on selectivity.

- ii. In yet another work by our group (Aba et al., 2013), it was shown that sorption efficiencies using groundnut and coconut shells based adsorbents in the uptake of lead ions (Pb^{2+}) from acid pickling effluent, that high percentage fixed carbon was responsible for better performance of coconut shell NaOH activated carbon in removal of 86.1% for Pb^{2+} .

3.3 DESIGN OF A PILOT PLANT TO TREAT BOTTLING COMPANY EFFLUENT

The major purpose of the breakthrough curve is to determine the time interval at which the used adsorbent must be regenerated (Fig 3.1). The breakthrough time here is 300mins.

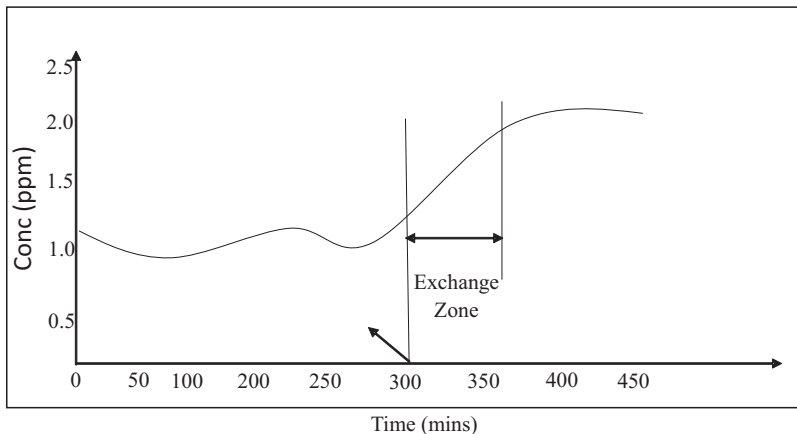


Fig 3.1 Breakthrough Curve

Source: Okafor and Aneke, 2006

3.3.1 EQUIPMENT VALIDATION

After 5 hours of operating column 3, it is stopped and regeneration begins in column 3, column 2 cools down while the process starts all over again in column 1.

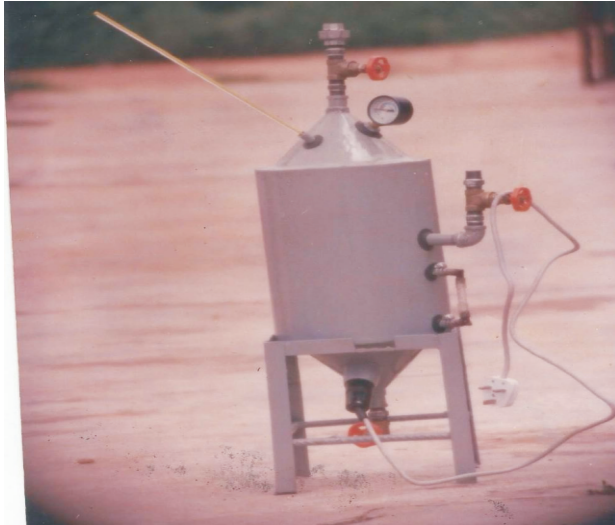


Fig 3.2 A steam chest

Source: Okafor, 2011

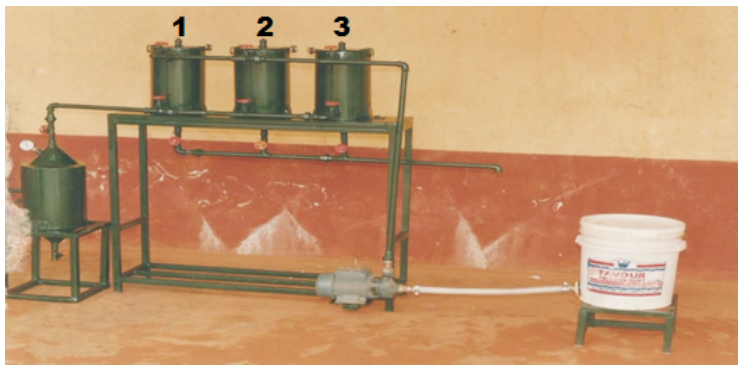


Fig 3.3 A process plant for purification of bottling company effluent.

Source: Okafor and Aneke, 2006

3.3.2 REGENERATION OF THE SPENT ADSORBENT

Regeneration was carried out in-situ using pilot plant of Fig 3.3. As the water boils, the steam valve is opened gradually to allow contact with the adsorbent. This was done for 1hr. The process of purification was repeated with regenerated adsorbents. Regeneration using steam here with the steam chest knocks out all particles (pollutants) blocking the pores of the adsorbents.

3.3.3 BENEFITS OF THE ADSORBER UNIT LOCALLY FABRICATED

The overall cost of materials and labour was N32,617.00 (Thirty-two thousand, six hundred and seventeen naira only). The result obtained after each regeneration (1st or 3rd) were subjected to a t-test statistical analysis. From the t-test analysis after regenerations, the adsorbents were as good as the fresh ones, which is quite commendable (Okafor and Aneke, 2006).

Again, the newly developed active carbons from cow bone have gone a long way to show that commercial industrial adsorbents can be replaced with ones from this work. This shall save several hundreds of billions of naira spent on importation of activated carbon in our Country.

3.3.4 EFFECTS OF CASSAVA EFFLUENTS FROM GARRI PRODUCTION

Table 3.3 Physical & Chemical Characteristics of Cassava effluents from some garri processing areas of Bida, Niger State, Nigeria.

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Table 3: Physical and Chemical Characteristics of Cassava Effluents from some garri processing areas of Bida, Niger State, Nigeria.

| Variables | Massaga | Efumayaki | Kotaworo | Edogifu | Efumadami | WHO(2004) Standard |
|------------------------------------|---------------------|---------------------|---------------------|---------------------|---------------------|-----------------------|
| Ph | 4.5 | 4.3 | 4.3 | 4.1 | 4.0 | 6.5 – 8.5 |
| Alkalinity (mg/CaCO ₃) | 150.0 | 140 | 141 | 130 | 129 | - |
| Conductivity (us/cm) | 40 | 50 | 52 | 41 | 61 | 750.5 |
| Temperature (°C) | 21 | 22 | 21 | 23 | 25 | 21 |
| Odour | Slightly Irritating | Slightly Irritating | Slightly Irritating | Slightly Irritating | Slightly Irritating | |
| *Cyanide (ppm) | 0.4 | 0.5 | 0.3 | 0.33 | 0.29 | 0.07* |
| BOD5(ppm) | 557 | 531 | 511 | 600 | 618 | 100 |
| COD (ppm) | 198 | 120 | 290 | 320 | 400 | |
| *BOD5/COD | 2.81 | 4.43 | 1.76 | 1.88 | 1.55* | |
| Dissolved oxygen (ppm) | 7.0 | 6.0 | 4.5 | 4.63 | 4.2 | 9.2 |
| Total Solid (ppm) | 483 | 420 | 512 | 470 | 700 | |
| Suspended Solid (ppm) | 221 | 183 | 210 | 194 | 315 | 25 |
| Dissolved Solid (ppm) | 262 | 237 | 302 | 276 | 385 | 1000 |
| Nitrate (ppm) | 0 | 0 | 0 | 0 | 0 | 50 |
| Hardness (ppm) | 600 | 320 | 550 | 481 | 420 | |
| *Colour (Hazen) | 23 | 21 | 25 | 22 | 23 | 5* |
| Chloride (ppm) | 120 | 107 | 100 | 130 | 160 | |
| Sulphate(ppm) | 0.54 | 0.50 | 0.49 | 0.50 | 0.43 | |

Source: Okafor, 2011

The WHO standard permissible limit for cyanide concentration is 0.07 ppm but for the five garri processing sites, they ranged from 0.29 to 0.5 ppm (76 – 86%) increase in Cyanide concentration. This is likely to trigger off environmental disaster in the immediate future. Another indicator of high environmental pollution is colour which ranged from 21 to 25 Hazen as against WHO permissible limit of 5 Hazen. According to George et al., (2003), the ratio of BOD/COD for an untreated waste water should be 0.3 – 0.8 but this study was from 1.76 to 4.43. This is high and unacceptable and above the admissible limit (Okafor, 2011).

3.3.5 ADSORPTION OF METRONIDAZOLE (MTZN) FROM WASTE WATER

Excessive use and improper disposal of waste pharmaceutical products such as analgesics, antibiotics, metronidazole (MTZN), hormonal drugs etc. have constituted a grave danger to our public health. Tea waste was carbonized at 400°C for 45 mins and modified with NaOH. The developed tea waste activated carbon (TWAC) was used as novel, cheap and eco-friendly adsorbent for MTZN removal. On doing BET and FT – Raman analyses, the BET surface area increased from 24.67 to 349m²/g, equilibrium time was attained after 75min. Linear and non-linear models were tested on the adsorption isotherm and kinetic data to determine the best fit for adsorption data. Freundlich isotherm and pseudo-second order models were found to be the best fit with a high correlation coefficient (R) of 0.999. The study on thermodynamics showed that the adsorption of MTZN was spontaneous, physical and exothermic in nature. Therefore, the non-linear models are more viable and powerful in describing the adsorption isotherm and kinetic data on the removal of MTZN onto TWAC according to our result (Ebili et al., 2020)

3.3.6 EFFECT OF ILLEGAL MINING

Illegal articial mining is rampant in Nigeria and has become a festering challenge both to governments and people living in the vicinity. In a study by our research group (Ibrahim et al., 2013) in Tunga Tsauni, Gurara L.G.A, Niger State, the water samples in these areas were heavily polluted with Pb^{2+} and therefore unsafe for human and animal consumption and aquatic biota. Other metals found in these samples within the sites include Zn(11.50mg/l), Pb (0.08mg/l), Cu (0.02mg/l), Cr (0.002mg/l) Fe (0.50mg/l), Mn(45mg/l) as against the NAFDAC permissible limit of 3.0mg/l, 0.01mg/l, 1.0mg/l, 0.05mg/l, 0.05mg/l and 0.7mg/l respectively. These revelations should help our governments of the day to take deliberate and concerted actions to secure our environment.

To control and predict the movement of these heavy metal ions (Pb^{2+} metal pollutants), we developed a mathematical model for prediction of pollutant movement. After testing, the tool was found capable of predicting lead mobility in the soil as to ascertain the safe areas for agricultural activities in Tunga Tsauni parts of Niger State. The correlation coefficients for these models are 0.99, 0.99, 0.99 and 0.99 along the West, North, South and East cardinal directions respectively in the illegal lead mining sites (Ibrahim et al., 2013).

3.4. CONVERSION OF WASTE PRODUCTS TO USEFUL PRODUCTS

i. Spent Engine Oil (SEO) is the oil removed from car/motor/truck engines done once in two or three months. These oils are carcinogenic and if discarded in farm lands, the land can hardly sustain living organisms. According to our earlier study (Ani et al., 2015), process parameters greatly influenced regenerating of base oil from SEO and the process is advantageous and of better quality oil and high quantity. In a similar vein, a mathematical model was developed which

explained the experimental data 99.96% and 99.14% for base oil and ash content respectively (Ani et al., 2015). However, heat to oil ratio has a synergistic effect on the base oil yield and ash content than time. The solvents can be recovered and reused which makes the process economically viable.

- ii. Waste cooking oil if allowed to accumulate in the environment shall in future become an enormous nuisance. Therefore our team of researchers with Abubakar et al., (2016), undertook a study on the optimization of biodiesel production from waste cooking oil. On using 24-factorial experimental design, we obtained an optimum yield of 90% of biodiesel from waste cooking oil. All values of process parameters from this work compare favourably well with the literature values and the standard biodiesel specifications.
- iii. Waste plastics are not only ubiquitous but in most instances are non-biodegradable. Some plastic waste materials disposed of in the last 50 years are still lying where they were dumped. For instance, the United States of America (USA) alone generates more than 170 million tons of plastic in a year. In one of our previous studies, we synthesized and characterized lubricant additives from plastic wastes. (Odigure et al., 2015). The waste plastics used were low density polyethylene, high density polyethylene, and polypropylene with optimum viscosity index of 162, 160, 159 and 186 corresponding to an increment of 80, 64-42, 36-76 and 76% for SN150, SN500, BS150, and YT3513 respectively. The lubricant additives synthesized from HDPE, LDPE and PP showed good pour points of -18, -20 and - 180°C respectively.

3.5 AIR POLLUTION ABATEMENT

- i. Air pollutants generated during cement manufacturing process consist primarily of particulates from the raw materials and finished products. This dust consists of oxides of Si, Al, Fe, Ca, K and S. A detailed investigation by our team on

the release of particulate pollutant concentration dispersed within Obajana cement plant in Nigeria showed that lower particulate concentrations were obtained in the year 2010 due to proper adoption of the control strategies and usage of high efficiency bag filters. Conversely, higher values of particulate pollutant concentrations were obtained for the year 2011. Results from Obajana cement plant show that this cement plant operates below the Nigerian acceptable limits on particulate matter concentration in Nigeria (Otaru et al., 2013), in WHO permissible limits.

- ii. Motor vehicle emission is a major source of air pollution in urban areas with CO as a major culprit, yet hardly any locally developed modeling tools are available for environmental protection agencies in Nigeria. A Gaussian Based Mathematical model was formulated for road traffic CO pollutant in Minna metropolis. The results obtained by us showed that the trend in modeled concentrations was similar to that of the measured concentration except for stability class D range the average value of index of agreement d , between the measured concentrations and the modeled concentrations was 0.89. the highest value of index of agreement obtained was 0.98 while the lowest value was 0.77. The model can be effectively used in predicting, monitoring and control of concentration of carbon monoxide pollutant in the atmosphere in Minna metropolis (Isa et al., 2013),
- iii. It was because of the desire to eliminate in the foreseeable future CO₂ from atmosphere that pilot plant study was carried out by our team to establish a base line assessment of CO₂ absorption with MEA at various concentrations with synthesized Mg-Co metal oxides (catalyst) from the exhaust fume of a power generating plant. Mg-O, CoO and MgO-CoO were prepared and tested. There was an increase in the yield of CO₂ captured when the metal oxides were used. The best yield of 11.9% was observed when 30g of MgO with 30%

concentration of MEA solution were used while MgO-CoO gave a yield of 9.9%. The percentage capture when only MEA solution was used was 4.1% showing that metal oxides enhanced the rate of carbon dioxide capture. Thus, the investigation serves as a green process that could be adopted to reduce global warming and guaranty safe and sustainable environment (Samuel et al., 2017). This process of carbon capture is called carbon sequestration.

3.6 LOCAL SHEA BUTTER BENEFICIATION IN NIGERIA.

- i. In a preliminary study of process route for quality improvement of traditionally produced shea butter our group of researchers investigated the various routes for the production of Shea butter to meet export requirement according to African standard for unrefined shea butter. The three traditional approaches namely sun drying, oven drying and manually operated rotary dryer gave in-consistent results that are below ARSUSB standards. However, some other parameters like saponification value (178.12-293mg KOH/g), free fatty acid (4.62 – 7.51%), iodine value (26.6 – 40.43mg/100g) and peroxide value (2.42 – 5.20mg/100g) were found to be at variance with ARSUSB standards. Percentage impurity (1.05 – 2.00%) is very much on the high side thereby necessitating further work to obtain good shea butter of industrial application (Saba et al., 2017),
- ii. Shea butter using traditional methods is currently not well standardized and therefore has negative significant impact on yield, product quality and pricing. We found that at 30°C roasting temperature, 31.68wt% Shea butter was recorded, which increased to 36.4wt% when the roasting temperature was raised to 150°C. FFA content increased significantly from 1.28 to 2.1 while PV was maximum at 100°C (2.1meg/kg). Longer roasting time and time lag between milling and kneading also led to high FFA and PV contents. Roasting

temperature, roasting time lag between milling and kneading represent the most significant parameters for high yield and good quality shea butter production (Saba et al., 2017) via traditional extraction method.

- iii. Therefore, the need to optimize these process variables becomes inevitable. The boundary conditions obtained from our preliminary studies were used for the design of experiment by Box-Bekehn method of response surface methodology. The optimization of these processing factors led to grade A shea butter at processing conditions of four days SNCP, 120min SNBD and 86°C of SNTD given shea kernel with 2.868mg/kg PV, 53.82% oil content, 0.628% FFA and a desirability index of 0.878. (Saba et al., 2019)
- iv. The traditional method involves numerous uncontrolled and non-specific practices. Other challenges affecting traditional approach includes hydrolytic oxidative and degradation of essential components, aflatoxin and attack of some micro-organism during post - harvest processing and storage consequently, shea butter produced with traditional method is characterized by high level of IV, high percentage of FFA and PV coupled with other solid and dissolved impurities. Consequently, therefore, we conceived the idea to design and to fabricate a rotary dryer for shea nut processing by the local processors in Nigeria to enhance the quality of shea butter product. The following process parameters can be put under control: temperature, moisture content, rain water not getting to the shea nut, drying time and production of shea kernel with uniform properties (Saba et al., 2018). Grade A shea butter is of high premium in shea butter international market. With this dryer in operation, grade A shea butter has been achieved.

3.7 MANAGEMENT OF AGRICULTURAL WASTES

3.7.1 Agricultural Solid Waste Conversion

- i. The process of producing carbon black for rubber reinforcement requires tremendous energy utilization, heavy infrastructural setup and constitutes a great source of environmental pollution because it is carcinogenic. We therefore, extracted nanoparticles from bamboo, coconut husk and cotton linter and characterised them on the basis of cellulose content and compared with commercial available N330 carbon black grade in terms of particle size distribution, particle topography and morphology as well as thermal stability. These biomasses were found to contain approximately 75,60 and 91% of cellulose for bamboo, coconut husk and cotton linter respectively. Dynamic light scattering (DLS) showed particle size distribution of 32-175, 68-311, 620-5400 and 150-385nm for bamboo, coconut husk, cotton linter and carbon black respectively. All the particles were found to be thermally stable beyond the processing and application temperature of most rubber composites. (Oboh et al., 2017).
- ii. We also evaluated the polydispersity index (PDI), it was found to be 0.371 far above 0.08 of the standard. Therefore, these cellulosic nanoparticles should be a suitable feed stock for rubber reinforcement at industrial scale (Oboh et al., 2018).
- iii. The tendency of cellulosic biomasses to absorb water, low thermal stability and poor compatibility with hydrophobic polymer matrix may pose limitations for their use on industrial scale. Coconut husk, bamboo and cotton linter cellulosic nanoparticles were used to reinforce neat rubber and the results compared with rubber reinforced with commercial available N330 carbon black in terms of thermal stability and thermo-molecular decomposition using TGA and differential scanning calorimeter (DSC) respectively. Our DSC results showed that cold crystallization and crystalline

melting temperature of rubber reinforced with 25 pphr of coconut husk, bamboo, cotton linter nanoparticles and carbon black occurred at temperatures of 67 and 358, 70.3 and 361.5, 70.5 and 365 and 70.4 and 365°C respectively while that of reference neat rubber was 65.9 and 354°C. The two temperatures compare favourably well (Oboh et al., 2019)

- iv. In yet another study by our team, we found that bamboo nanoparticles were polydispersed with size distribution of 32-175 nm, the tensile strength of the composite produced with bamboo based nanoparticles increased from 1.8 MPa for neat rubber vulcanizate to 3.16 MPa with the incorporation of bamboo based cellulosic nanoparticles into the rubber matrix as the nanoparticle was increased upto 30 pphr. (Oboh et al., 2017). The TGA study showed onset decomposition temperatures of 216, 252 and 256°C for unfilled rubber, NR-BNC and BNC respectively. These results are quite encouraging because there was good interfacial interaction between rubber matrix and the bamboo based cellulosic nanoparticles evident in the increase of tensile strength and reduction in number and sizes of pores present in the unfilled rubber matrix.
- v. To elucidate further the thermal, morphological and mechanical properties of composites from bamboo, coconut husk, cotton linter and N330 carbon black were evaluated. We have shown through SEM study that the free volume holes in the neat rubber were drastically reduced by incorporation of these nanoparticles in to the rubber matrix. Coconut husk based composites showed a trend of increase in tensile strength from 1.8 to 3.82 MPa with filler loading of 0 to 25 weight percent, while bamboo, cotton linter and carbon black based nanoparticles have their values of 3.16, 3.92 and 4.50 MPa respectively at filler content of 30 weight percent.
- vi. The composite we obtained by reinforcing neat rubber with these nanoparticles as well as NR-CB were subjected to DSC,

and again we have shown that all samples of composites have similar curves. These curves showed both negative region (an exothermic event eg crystallization) and a positive region (an endothermic event e.g melting). See figure 3.4 (Oboh et al., 2017).

3.7.2 Thermal properties of composites

3.7.3 Differential Scanning Calorimetry (DSC).

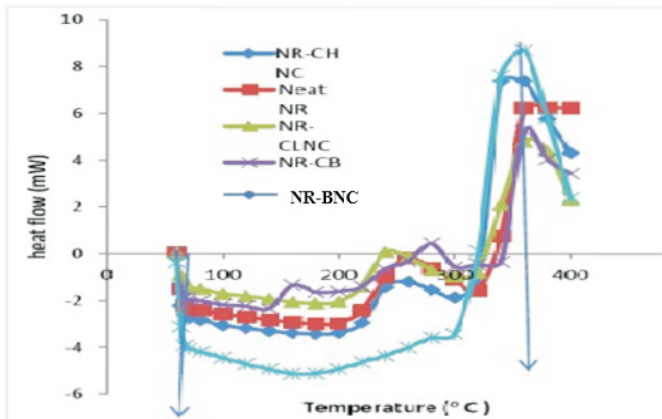


Fig 3.4 Effect of filler types on DSC curves of composite.
(Source: Oboh et al, 2017)

The process of crystallization is exothermic, hence differential heat input will be negative. From the results obtained, the cold crystallization temperatures (T_{cc}) for the various samples were found to be 65.9, 67, 70.3, 70.5 and 70°C for neat NR, NR-CHNC₂₅, NR-BNC₂₅, NR-CLNC₂₅ and NR-CB₂₅ respectively.

On completion of crystallization, a new baseline which relates to the specific heat capacity of the polymer samples will be established. As temperature is further increased, there will be little or no change in differential heat input until the crystallization melting temperature (T_m) is reached, at this

point the polymer crystals begin to melt, a process that is endothermic and signified by a positive change in differential heat input. The crystallization melting temperatures (T_m) were found to be 354.4, 358, 361.5, 365, and 365 °C for neat NR, NR-CHNC₂₅, NR-BNC₂₅, NR-CLNC₂₅ and NR-CB₂₅ respectively. In addition to T_{cc} and T_m , the degree of crystallinity of the neat rubber and composite samples were calculated using Equation 3.4 (Olatunji and Richard, 2016)

$$X_c = \frac{\Delta H_m - \Delta H_{cc}}{(1-f)\Delta H_M} * 100\% \quad 3.4$$

where X_c is the degree of crystallinity of composites, ΔH_{cc} and ΔH_m are crystallization and melting enthalpy changes respectively and were obtained from DSC curves, f is the weight fraction of the fillers (25 pphr or 20%), Table 3.4 shows the degree of cryatallinity (X_c) of samples and other thermal characteristics obtained from DSC, according to our findings shown on table 3.4 (Oboh et al., 2017).

Table 3.4: Crystallinity and melting characteristics of composite samples

| Samples | H_{cc} (J/g) | H_M (J/g) | f | T_{cc} (°C) | T_M (°C) | X_c (%) |
|-----------------------|----------------|-------------|------|---------------|------------|-----------|
| Neat NR | -3.908 | 0.298 | 0 | 65.9 | 354.4 | 21.62 |
| NR-CHNC ₂₅ | -2.692 | 8.111 | 0.20 | 67 | 358 | 43.26 |
| NR-BNC ₂₅ | -4.056 | 8.595 | 0.20 | 70.3 | 361.5 | 36.24 |
| NR-CLNC ₂₅ | -1.432 | 4.811 | 0.20 | 70.5 | 365 | 26.97 |
| NR-CB ₂₅ | -1.947 | 5.77 | 0.20 | 70.4 | 365 | 30 |

Source: Oboh et al., 2017

In all cases, the inclusion of 25 pphr (20 % wt) fillers into rubber matrix increased the cold crystallization temperature (T_{cc}), crystalline melting temperature (T_m) as well as the crystallinity (X_c) of the resultant composites. This could be due to the

crystalline nature of cellulosic materials as well as carbon black. One of the striking interests revealed by the outcome of this DSC analysis was the correlation between the calculated crystallinity and tensile strength of the cellulosic particle based composites. Recall that tensile strength values of cellulosic based composites occurred in descending order of 3.82, 2.82 and 2.62 MPa for NR-CHNC₂₅, NR-BNC₂₅ and NR-CLNC₂₅ respectively; this same trend was repeated in the case of their degree of crystallinity, such that 46.26, 36.24 and 26.97% were for NR-CHNC₂₅, NR-BNC₂₅ and NR-CLNC₂₅ respectively.

3.7.4 Thermo-Gravimetric Analysis (TGA) OF COMPOSITES

Thermo gravimetric analysis (TGA) of composite sample was conducted in order to determine thermal stability by measuring the change in the weight of the composite samples when heated in air. Figure 3.5 shows the effect of filler types on TGA curves.

Three broad thermal regions are usually associated with TGA/DTG, such that first, second and third regions correspond to drying/ evaporation, decomposition /devolatilization and complete degradation respectively. The first region is used to determine the onset of thermal degradation temperature which is defined as the temperature at which sample losses are not more than 5 % of its initial mass. Other important parameters that can be obtained from the TGA/DTG curves are $T_{50\%}$ (temperature at which the sample losses 50% of its initial mass), T_{max} (temperature at which maximum rate of degradation obtainable from DTG curves) and percentage inorganic residues are shown in Table 3.4.

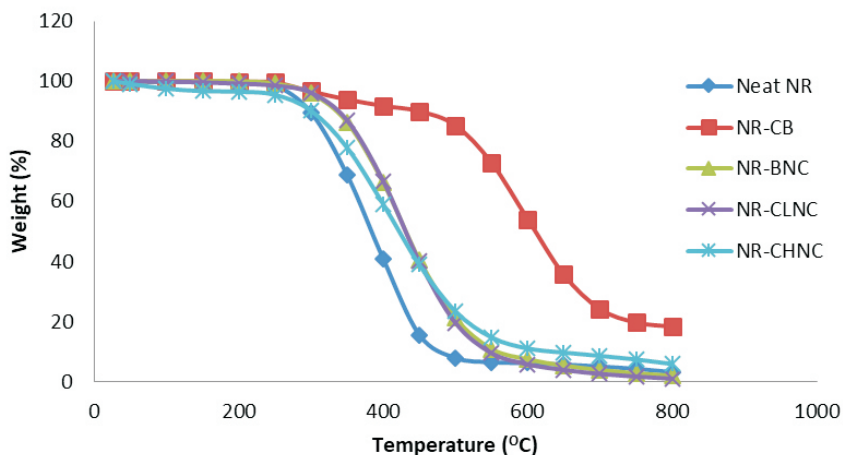


Figure 3.5: Effect of filler type on TGA curves of composites

Source: Oboh et al., 2019

NR and composites reinforced differently with carbon black, bamboo, coconut husk, cotton linter cellulosic nanoparticles showed an initial weight loss of 0.1-03% at about 70-100°C. The onset of thermal degradation was 266°C for NR and shifted toward 299, 300, 308 and 240°C upon incorporation of bamboo, cotton linter, carbon black and coconut husk nanoparticles respectively into the rubber matrix. From our research findings, this again shows that these nanoparticles can replace or serve as alternative materials to carbon black in rubber industries (Oboh et al., 2017).

3.8 CONCLUSION

This lecture has shown that:

- i. Bone derived active carbon can be used in Bottling company effluent and that offensive odours in bottling company effluents were efficiently removed
- ii. The pilot plant that was designed and constructed is functional and is capable of purifying bottling company effluents to WHO admissible limits of effluent discharge into

the environment. This can be modified to treat other liquid effluents from small scale, medium scale and large scale industries in Nigeria.

- iii. Grade A shea butter at processing conditions of 4.33 day of SNCP, 10min SNBD and 86.23°C of SNTD resulted to shea butter of 2.869meq/kg of PV, 0.628% of FFA, percentage oil content of 53.58% and desirability index of 0.878
- iv. A rotary shea nut dryer was designed and fabricated and tested. The dryer was found to put some important process parameters under control.
- v. Tensile strength and other physico-chemical properties increased with inclusion of cellulosic nano particles into the neat rubber matrix.
- vi. The physico-mechanical properties of these composites compared favourably well with rubber matrix reinforced with carbon black and therefore could be replacement for carbon black.
- vii. The final product has an enhanced aesthetic value whereas previously rubber products are black but this time around the colour ranges from grey white, light yellow and light brown.

3.9 RECOMMENDATIONS

- i. Process intensification of designed and constructed pilot purification plant.
- ii. Design and fabrication of a kneading machine with appropriate speed, direction of rotation and impeller type to improve recovery of shea butter from the shea paste.
- iii. Investigate the effect of shea nut variety on the optimization parameters of both the shea kernel and the extraction method.
- iv. The use of compatibilizer or surface functionalization of cellulose nanoparticles.
- v. The hybrid effects of more than one cellulosic nanoparticle type on natural rubber matrix.

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University Management

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Department of Chemical Engineering.

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Vice-Chancellor Sir, I am done.

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THE BRIEF PROFILE OF THE INAUGURAL LECTURER

Engr. Professor Joseph Onyebuchi Udeagbala Onwuegbuchunam Okafor was born on 8th August, 1956 into the family of late Ogbuefi Isaac Ezejiofor Okafor and Owanwanyi Mary Uzoigwe Okafor in Owelli, Enugu State.

Prof. Okafor started his educational pursuit at St. Patrick's Catholic Primary School, Ogbete, Enugu and later continued at St. Patrick's Catholic Primary School Iva-Valley, Enugu. At the end of Nigerian-Biafra hostilities, he finished his primary school career at St. John's Catholic Primary School, Owelli in Awgu Local Government Area of Enugu State. He went to St. Vincent's Secondary School, Agbogugu, also in Awgu L.G.A.

On completion of his secondary education, Prof. Okafor travelled to Romania where he did B.Sc Chemical Engineering. On return to Nigeria, he did his National Youth Service Corp (NYSC) at the Kaduna Polytechnic in Science Technology Department. He finished precisely in October, 1984. The then Anambra State Government employed him to serve with Secondary School Management Board and later moved to Institute of Management and Technology (I.M.T) Enugu. Throughout his stay at I.M.T, he was the examination officer of the Department of Chemical Engineering.

He joined Federal Polytechnic, Bida in 1996, and was appointed the Examination Officer of the Department of Chemical Engineering. He kept a record of longest serving Examination Officer. (1997 - 2005). Prof. Okafor had the privilege of serving as the Head of Chemical Engineering Department, Federal Polytechnic Bida for three terms.

Prof. Okafor saddled with administrative and academic responsibilities never relegated his personal development to the background and by 2006, he was appointed Chief Lecturer of Chemical Engineering. While at the Federal Polytechnic, Bida, he attended several professional, University and International conferences ditto for his journal publications. Other positions handled by Prof. Okafor while at the Polytechnic include; Zonal Coordinator, Nigerian Society of Chemical Engineers (Bida);

Chairman, Committee on Science and Technology Education Post-Basic (STEP-B) Project, Federal Polytechnic, Bida; Resource person, National Board for Technical Education (NBTE) for Re-Accreditation/Resource Inspection of Chemical Engineering programmes in Polytechnics.

After eleven blissful years at the Federal Polytechnic, Bida, Prof. Okafor, desired to have a change of environment and therefore applied for a job at Federal University of Technology, Minna, in the Department of Chemical Engineering. He was offered the job and by January, 2008, he assumed duty. As usual, he was made the Examination Officer, but this time, the appointment was short lived (January 2008 to September, 2008).

However, Prof. Okafor served and is still serving in other capacities in the Department and the University. As the interim H.O.D of Chemical Engineering, Prof. Okafor coordinated the movement of Chemical Engineering Department from Bosso to Gidan Kwano. He was the Chairman, Consultancy Services, (Department of Chemical Engineering); he was the Departmental Post-Graduate (PG) Coordinator; Chairman, Departmental Design Project Committee; Chairman, Committee on Book Development; Editorial Board Member, Nigerian Journal of Engineering and Applied Sciences, FUT Minna; Member, Technical Committee on Centre for Shea Research and Development.

Prof. Okafor has had the opportunity to assess the works of some colleagues for promotion to Chief Lecturer and to professorial cadre and also an external examiner to some Departments of Chemical Engineering in Polytechnics and Universities. He reviews journal papers for at least four publishing houses.

Prof. Okafor has published copiously in international journals and journals within the shores of Nigeria. He is a Registered Engineer (R.Eng).

It might interest you to know that Prof. Okafor (Principal Investigator) with his team of scholars recently won a TETFund National Research Fund Intervention worth Twenty-Five Million, Three Hundred and Fifty-Five Thousand, Five Hundred Naira Only (N25,355,500). Engr. Prof. Joseph O. Okafor is married with children.