STUDIES ON THE IMPACT RESISTANCE OF CASHEW NUTSHELL POWDER AND CALCIUM CARBONATE FILLED POLYPROPYLENE

BY

SHU'AIBU MUSA ABUBAKAR

DEPARTMENT OF CHEMISTRY
FACULTY OF SCIENCE
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA

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BY

Shu'aibu Musa ABUBAKAR, B. Sc CHEMISTRY (A.B.U), 2010
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DEPARTMENT OF CHEMISTRY,
FACULTY OF SCIENCE
AHMADU BELLO UNIVERSITY, ZARIA
NIGERIA

APRIL, 2016

DECLARATION

I, Shu'aibu Musa ABUBAKAR, declare that this dissertation entitled "STUDIES ON THE IMPACT RESISTANCE OF CASHEW NUTSHELL POWDER AND CALCIUM CARBONATE FILLED POLYPROPYLENE" has been carried out by me in the Department of Chemistry. the information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this thesis are was previously presented for another degree or diploma at this or any other Institutions.

Shu'aibu Musa ABUBAKAR (P13SCCH8031)	**Signature**	**Date**

CERTIFICATION

This is to certify that, this dissertation entitled "STUDIES ON THE IMPACT RESISTANCE OF CASHEW NUTSHELL POWDER AND CALCIUM CARBONATE FILLED POLYPROPYLENE" by Shu'aibu Musa ABUBAKAR meets the regulations governing the award of the degree of MASTERS DEGREE IN POLYMER SCIENCE AND TECHNOLOGY of the Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

Chairman, Supervisory Committee (Dr. P.A.P MAMZA)	**(Signature)**	**Date**
Member, Supervisory Committee (Dr. H. ABBA)	**(Signature)**	**Date**
Head of Department (Prof. V.O. AJIBOLA)	**(Signature)**	**Date**
Dean, School of Post Graduate Studies (Prof. K. BALA)	**(signature)**	**Date**

DEDICATION

This research is dedicated to the creator and sustainer of the universe, Almighty Allah and the entire family of Mallam Abubakar Musa Kudan who, through their support and encouragement, I was able to successfully complete this research and enjoy my stay in the university environment.

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I am indebted to my Father (Mr. Abubakar Musa) and Mother (Mrs. Mariya Shuaibu) for their unconditional love and support. Without their help, it would not have been possible to complete my wonderful journey. I bow my head in respect for all that they have done for me. I also wish to express my gratitude to my colleague Mr. Abdul-wahab I. Okele for giving me moral support of most of the technological aids. I share this happy moment with my friend M.B Shamsu Miruwa for rendering me enormous support during the whole tenure of my research.

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ABSTRACT

Mechanical and morphological properties of pure polypropylene (PP) polypropylene/calcium carbonate (PP/$CaCO_3$) and polypropylene/cashew nutshell powder (PP/CNSP)are reported in this work. The composites were prepared by compression moulding technique. The compressed moulded articles that is the PP, (PP/$CaCO_3$) and (PP/CNSP) of different compositions (10/90, 20/80, 30/70, 40/60, 50/50, 60/40, 70/30, 80/20) were characterised for mechanical properties, water absorption capacity, structural characterisation and morphological arrangements. Comparative studies was made on the mechanical properties of the pure polypropylene (PP), polypropylene/calcium carbonate (PP/$CaCO_3$) and polypropylene/cashew nutshell powder (PP/CNSP). Mechanical properties such as tensile strength, Young's modulus and percentage elongation at break, Hardness behaviour and Impact resistance of both PP/$CaCO_3$ and PP/CNSP composites increased with increment of filler weight content (10-50g). It was noted that the specimen samples of ratio50/40 PP/$CaCO_3$ and PP/CNSP had the highest tensile strength, when compared with other sample. These specimens could bear loads of 1075N and 468N with extensions of 4.44mm and 6.12mm respectively. Decrease in the mechanical properties were noted on continuous addition of both fillers, with drastic reduction of the mechanical properties at (70g and 80g) fillers weight except hardness that slightly increased at all the filler loading (10-80g). The surface sorption characteristics of calcium carbonate and cashew nutshell powder have been investigated and the highest percentage was recorded at 20/80 of PP/CNSP (100%). Scanning electron microscopy (SEM) revealed that, both 60/40 PP/$CaCO_3$, PP/CNSP and 50/50 PP/$CaCO_3$, PP/CNSP are completely compatible at which there are no phases that are grossly separated. X-ray diffraction analysis showed that, the incorporation of the two fillers into the neat

polypropylene decreased the crystallinity of the polypropylene and the crystallinity decreases with increasing filler's loading.

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LIST OF ABBREVIATIONS

ABS	Acrylonitrile-butadiene-styrene
AFM	Atomic Force Microscope
ASTM	American Society for Testing Materials
$CaCO_3$	Calcium Carbonate
CNSP	Cashew Nutshell Powder
CRH	Chopped Rice Husk
EVA	Ethylene vinyl acetate
HDT	Heat Distortion Temperature
HPP	homogeneous polypropylene
IM	Initial modulus
LDPE	Low density polyethylene
LLDPE	Linear low density polyethylene
LOE	Linseed Oil Epoxide
NR	Natural Rubber
OM	Optical Microscope
PE	Polyethylene
PEMA	Poly (ethyl methacrylate)
PET	Polyethyleneteraphthalate
PIB	Polyisobuthylene
PMMA	Polymethamethylacrylate
PP	Polypropylene
PPC	Polypropylene copolymer

PRPCs	Particle reinforced plastics composites
PS	Polystyrine
PVAc	Polyvinyl acetate
PVC	Polyvinylchloride
SALS	Small angle light scattering
SAXD	Small-Angle X-ray diffraction
SAXS	Small-Angle X-ray Scattering
SD	Spinodal decomposition
SEM	Scanning Electron Microscopy
TS	Tensile strength
UTM	Universal Testing Machine
WAXD	Wide Angle X-ray Diffraction

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Particle reinforced plastics composites (PRPCs) are composites to which fillers (discrete particles) have been added to modify or improve the properties of the matrix and/or replace some of the matrix volume with a less expensive material. Common applications of PRPCs include structural materials in construction, packaging, automobile tires, medicine, etc. Determination of effective properties of composites is an essential problem in many engineering applications (Van, 2003 and Love, 2004).

These properties are influenced by the size, shape, properties and spatial distributions of the reinforcement (Liu, 1995 and Lee, 1998).

Modification of organic polymers through incorporation of additives yield, with few exceptions, multiphase systems containing the additive embedded in a continuous polymeric matrix. The resulting mixtures are characterised by unique microstructures that are responsible for their properties. Polymer composites are mixtures of polymers with inorganic or organic additives having certain geometries. Thus, they consist of two or more components and two or more phases. In addition to polymer composites, other important types of modified polymer systems include polymer-polymer blends and polymeric forms. Blending procedures had been employed since time immemorial. The principle of blending is geared towards achieving property averaging. A blend is therefore the physical mixture of two or more substances, without a chemical bond, (Mamza, 2011).

Among the various studies carried out with particle filled PP worth mentioning, are works by Maiti and Mahapatro (1992 and 2011) on the tensile and impact behaviour of nickel powder-filled PP and $CaCO_3$ filled PP composites. It was discovered that the addition of nickel-powder causes decrease in tensile modulus, tensile strength and elongation-at-break with increasing filler. In the case of the addition of $CaCO_3$, tensile modulus increased while tensile strength and elongation-at-break decreased with increasing filler. Izod impact strength for the composites at first application of filler loading increased up to a critical filler content, beyond which the value decreased inappreciably.

1.2 Research problem

The filler cashew nutshell powder (CNSP) has been under utilised, in composite formulation, as it is considered as waste material especially in the Northern part of Nigeria. Thus, there is need to convert this waste to wealth meanwhile this conversion would serve as an environmental waste control.

1.3 Aim and objectives

The main aim of this work was to determine the impact resistance of cashew nutshell powder and calcium carbonate used as fillers for polypropylene.

The specific objectives of the study are;

1. Collection of samples from the outlet centre and preparation of samples.
2. Determination and characterisation of cashew nutshell powder using X-ray diffraction analysis.
3. To carryout mechanical tests such as hardness, tensile strength, elongation at break, impact resistance and to carry out sorption test on the produced samples,

4. Determination of microstructure of the processed samples using scanning electron microscopy (SEM).

1.4 Justification

Cashew nut shell powder as one of the fillers used in this research can reduce the cost of production of articles compared to the commercially available fillers. It can create job opportunity locally, by paying people supplying it for the researchers. The use of cashew nutshell powder as filler can help to reduce environmental pollution caused by the shell, this is because, it is biodegradable and it can decay and becomes a pollutant to the society.

1.5 Scope of the study

1. To prepare and characterise CNSP filler
2. To fill cashew nutshell powder in polypropylene
3. To fill calcium carbonate in polypropylelene
4. To carry out mechanical tests on the prepared composite, such as hardness, tensile strength, elongation at break, sorption test, and impact resistance
5. To compare the impact resistance of cashew nutshell powder and calcium carbonate filled polypropylene

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Background of Literature

Reinforcement of polymer blends is particularly useful in those cases where blending produces improvement of some properties with simultaneous determination of other properties such as mechanical strength, modulus, stiffness, etc, thus in order to retain the advantage of blending, it becomes necessary to compensate for the loss of properties by reinforcement with suitable fillers (Gupta, 2002). Properties of a given polymer can be improved in various ways such as blending with other polymers or by incorporation of reinforcing fillers (Mamza, 2011).

Mamza and Folaranmi (1996) carried out compatibility studies on solution of polystyrene (PS) and poly(vinyl acetate) (PVAc) blend by density and viscometric methods. The studies revealed that the experimental densities of the blends were found to be lower than the calculated values assuming additivity of volumes of polymers and solvent, while the plots of relative viscosity with composition were of the S-type, indicating two-phase formation with phase separations at intermediate compositions and the comparison of the calculated (using the Krigbaum – Wall Equation for both solvents) and observed intrinsic viscosities showed higher calculated values, thereby suggesting that PS and PVAc are an incompatible pair.

Nwufo, (Griffin *et al.*,1984) studied extrusion of starch-extended water-soluble poly(vinyl Alcohol) and asserted that the method is relatively faster in estimating the physico-mechanical properties of the virgin polymer and its composite, thereby enabling the prevention of the extrudate so produced during extrusion from water effects. While studying the microscopic arrangement/appearance of fractured surfaces and some mechanical properties of starch-

extended poly (vinyl alcohol), Nwufo and Griffin (1985), with the aid of scanning electron microscopy (SEM), showed that the composite with 0% starch content reflected only fracture lines, whereas composites with 10% w/w starch content in Mowiol (an industrial plasticized poly (vinylalcohol) showed fracture lines as well as some pitting, indicative of starch beds. The mechanical properties of the composites revealed that the impact strength decreases with increase in starch content, in comparison to the matrix impact property. It was explained that the swelling of starch in the matrix brings about a further increase in the percentage of starch resulting in a break, however, the breaking strength decreased progressively with increase in the starch content. Also, the flexural modulus showed increase with increase in starch content.

Olawole and Agboola (1982) conducted some mechanical property studies on polystyrene (PS) and its blends with acrylonitrile-butadiene-styrene, PVC and polybutadiene and observed an increase in modulus with exposure time while the elongation decreases.

Olayemi and Ibiyeye (1986) evaluated the mechanical properties of blends of poly (vinyl acetate) and poly (ethyl methacrylate) and observed improvements in the mechanical properties of the polymers due to blending. These were considered to be as a result of the presence of favourable and strong (PVAc – PEMA) intermolecular interactions which revealed miscibility and compatibility of the polymers. In this study, a lower critical value (Mv) of 4.9 was found, above which phase separation would be expected on blending these two polymers to the extent of 20% weight of PEMA. The stress-strain curves for the PVAc and PEMA samples and some of their blends indicated a somewhat rubbery, soft and weak characteristic of PEMA as the percentage of the latter was increased in the blend. Also, the dependence of tensile strength (TS) of films of the polymers on blend composition, showed an increase from that for PVAc with a peak, followed by a decrease to a minimum, then a final increase to the value for PEMA, with increase in

percentage PEMA, in the blend. Some areas above and below the additivity straight line joining (TS) PVAc to (TS) PEMA were observed in the study. However, they concluded that 20% PEMA produced the optimum effect on TS, initial modulus (IM) and % elongation at break of the films obtained from the blends. Films from blends with up to 30% PEMA, and, in particular, those with 20% PEMA, had high values of TS, IM and % elongation at break, and are, on the basis of the mechanical properties studied, significantly superior to the individual polymers that constitute the blends.

Folaranmi and Zayyan (2002) studied some properties of polystyrene (PS) – polyisobutylene (PIB) rubber blends and observed that at higher concentrations of PIB in the blend the transparency was impaired and phase separation was observed. The results of the mechanical test on some selected films showed a higher value of yield stress and ultimate strain and a larger area under the stress-strain curve for blends than for PS, suggesting that PS was toughened by blending with the PIB rubber samples. They observed the highest value of Young‟s modulus for 95/5 blend with subsequent decrease for blends with higher rubber content. It was concluded from the study that PS was semi-compatible with PIB at low percentage concentration of rubber in the blend and at low solid content of solution. There was also a tendency of the blend to demix at higher rubber content of the blend as reported from the film morphology study. Toughening of PS with PIB rubber must therefore be at fairly low rubber concentrations, preferably < 10% of the total weight.

Mamza and Folaranmi (1996) carried out compatibility studies on solution of polystyrene (PS) and poly(vinyl acetate) (PVAc) blend by density and viscometric methods. The studies revealed that experimental densities of the blends were found to be lower than the calculated values assuming additivity of volumes of polymers and solvent, while the plots of relative viscosity

with composition are of the S-type, indicating two-phase formation with phase separations at intermediate compositions and the comparison of the calculated (using the Krigbaum – Wall equation for both solvents) and observed intrinsic viscosities showed higher calculated values, thereby suggesting that PS and PVAc were an incompatible pair.

(Ashraf *et al.*,2007) studied and highlighted the utilization of linseed oil epoxide (LOE) (a product from a sustainable resource), to obtain blends of LOE with polystyrene (PS) forming tough and flexible free-standing films. The mechanical properties of LOE/PS blend films were found to match with those of LDPE at composition LOE/PS, 65/35. The potential applications for such sustainable resource-based blend included packaging films and production of biodegradable plastic sheets which can be formed into products such as bio-bags. It was observed in this study that the blends of LOE with PS were miscible in solution phase in the composition range of LOE/PS, 85/15 to 45/55 as confirmed by viscosity and density measurements. At composition LOE/PS, 35/65 phase separation took place which indicated the onset of immiscibility. The morphological investigation revealed a two-phase system in the case of LOE/PS, 85/15. The toughness of the films was found to increase with increasing content of PS in the blend. The mechanical properties of LOE/PS blend films were found to match with LDPE at composition LOE/PS, 65/35. The study concluded that the linseed oil epoxy (a product from sustainable resource) can be substituted up to 65% or even higher with Polystyrene to obtain tough and flexible films. Gupta and Singh, (2005) characterized by spectral, structural, thermal and electrical methods, polymer composites of O- tolidine-iodine (1:0.75 molar ratio) charge-transfer complex with polystyrene prepared in different weight ratios. The polymer composites exhibited semi-conducting behavior. The current-voltage characteristics, frequency dependence of conductivities of these composites were determined. The frequency dependence

of ac conductivity of these composites showed contribution from grain, grain boundary and electrode as a function of charge-transfer content. They observed that at low charge-transfer content, all the three contributions were present but at high content, only grain contribution was relevant.

Chemorheological analysis of the modified network was performed to understand the physical transformation taking place during the cure polymerisation reaction. The delay in gel time on inclusion of rubber was explained by lower reactivity due to dilution and viscosity effect.

Tensile, flexural, and fracture toughness behavior of neat as well as modified networks have been studied in order to observe the effect of rubber modification. The morphological evolution of the toughened networks was examined by scanning electron microscope, and the observations were used effectively to explain the impact properties of the network having varying content of liquid rubber. Also, acoustic emission studies were performed on neat and certain modified systems. Based on acoustic emission results and morphological characteristics, toughening and failure mechanisms were discussed. The behaviour of the relaxation peaks were evaluated by dynamic mechanical analysis; the researchers also tried to explain the composition of networks. The thermal stabilities of the toughened epoxies were studied using thermogravimetric analysis (TGA). From this study, the activation energy for decomposition of neat and modified epoxies was estimated and compared. It was concluded that the reduction in cross-linking density of the thermoset upon modification can be confirmed and explained.

Folaranmi, *et al.*, (2001) reported the effect of polyisobutylene rubber concentration and molecular weight on density and relative and intrinsic viscosities of PMMA/PIB blend solutions in toluene, as well as the morphology and tensile mechanical properties of some of the blend films.

Also, clay incorporation and peroxide treatment did not significantly change the tensile properties.

Ahmad, *et al.*, (2004) assessed the effects of filler (carbon black N110, silica and calcium carbonate) on the mechanical properties of natural rubber (NR)/Linear low – density polyethylene (LLDPE) blends. The mechanical and physical properties of the blends were found to be dependent on the filler characterization. Swelling index and elongation – at – break showed a decreasing trend with increasing volume percent of filler loading in the blend. The significant changes in other physical properties included the gel content, and bound rubber of the samples indicated a strong interaction between the filler particles and polymer matrix. In conclusion, mean agglomerate particle size and polymer-filler interaction were the factors determining the mechanical and physical properties of NR/LLDPE blends. The reduction of elongation-at-break were due to stiffening of the polymer matrix by the filler. Further increase in filler loading causes the molecular mobility decreased due to extensive formation of physical bonding between the filler particles and the polymer chain that stiffened the matrix. A progressively reinforced and hence lowered elongation –at-break at any filler loading greater than 20% was observed. They observed also that with good polymer-filler interactions, there would be increase in modulus as well as mechanical properties.

(Razavi-Nouri *et al.,* (2006) reported the mechanical properties and water absorption behaviuor of chopped rice husk-filled polypropylene composites. In the study the reinforcing effect of chopped rice husk (CRH) in polypropylene was studied. Composites containing different amounts of CRH with 0 to 40 parts per hundred parts of polymer (php) were prepared using a co-rotating twin screw extruder and characterized by determination of their mechanical properties and also water absorption. In order to increase the interphase adhesion, polypropylene grafted

with maleic anhydride was added as a coupling agent to all the composites studied. It was found that the tensile and flexural modulus of the composite containing 40php of CRH increased approximately by 33% and 100%, respectively. The results also showed that, while flexural strength was moderately improved by increasing of CRH into the matrix, elongation-at-break and energy-at-break decreased dramatically. A reasonable adhesion between the main components was also observed in the spectra of the scanning electrons micrographs. Water absorption experiments, showed that although the diffusion coefficient increased with CRH loading, all the composites followed case diffusion.

From their study, it was observed that the detrimental effect of CRH addition into PP containing MAPP (the matrix) became apparent such that of CRH reduced the ductile behaviour of the matrix by making the composites more brittle. While the matrix showed ductile behaviour via a neck formation, the composites consisting CRH revealed brittle behaviour with no necking. Although the tensile strength of a composite was more sensitive to the matrix properties, its modulus was dependent on the fibre properties. To improve the tensile strength, a strong interface, low stress concentration and fibre orientation were required, whereas high fibre aspect ratio and fibre wetting determined tensile modulus. The impact strength showed a slight decrease in value after addition of 10 phr of CRH into the matrix. However, it leveled off when higher amount of CRH was added into the composite. The ductile deformation of the matrix was inhibited because of the presence of CRH. Three different mechanisms have been proposed for moisture penetration into the composite and the main process is the diffusion of water 80 molecules into the composite, while the main process is the diffusion of water molecules inside the microgaps between the polymer chains. The other two mechanisms are capillary transport of water into the gaps and flaws created at the interface of fibre and polymer matrix, because of

incomplete wettablility and impregnation and also diffusion of water molecules into the microcracks formed in the matrix during the compounding process (Lin, Q. 2002).

Mishra and Luyt, (2008) reported the effect of organic peroxides on the morphological, thermal and tensile properties of EVA – organoclay nanocomposites. They observed a flocculated morphology and reduced Polymer clay interaction from the prepared nanocomposites. There is a good correlation between these morphologies and the thermal stabilities and total crystallinities of the nanocomposites.

Thomas *et al.,* (2008) studied the miscibility, morphology, thermal and mechanical properties of DGEBA-based epoxy resin toughened with liquid rubber. In the study, epoxy resin based on diglycidyl ether of bisphenol A and varying content of hydroxyl terminated poly(butadiene) was cured using an anhydride hardener. The ultimate aim of the present study was to modify the low Impact matrix by cashew nutshell powder and calcium carbonate which is a commercially available filler to improve the toughness characteristics.

There have been many studies using nano calcium carbonate to enhance the properties of polymers. Among the techniques employed to disperse the nano filler include in-situ polymerisation, melt mixing using internal mixers and melt compounding using twin-screw extruders. In-situ polymerization technique has been used for PVC and PET, while melt mixing and melt compounding appeared to be the preferred method for polypropylene (Ritchie, 1993).

Eiras and Pessan (2009) presented a paper on the effects of calcium carbonate nano particles in the crystallization of polypropylene. The experimental work included Differential scanning calorimetry analysis of isothermal and nonisothermal crystallization, optical microscopy and X-ray diffraction. In their study, four compositions of PP/$CaCO_3$ nano composites with calcium carbonate content of 3%, 5%, 7%, and 10% by weight were prepared in a co-rotational twin

screw extruder machine (Weiner and Pfleirer ZSK-30) with temperature profile of 170/190/190/190/190/1950C, and a screw speed of 100rpm. DSC analysis were conducted by heating the samples from 300^{o}C to 2000^{o}C at a heating rate of 100C/min keeping the sample at this temperature for 2 minutes and then cooling down from 2000^{o}C to 300^{o}C at a cooling rate of 100C/min. The isothermal analysis and optical microscopy analysis were conducted by heating the sample at the same condition and then cooling from 2000^{o}C to their crystallization temperature, maintaining it at this temperature for 15min.

X-ray diffraction analysis was conducted in a Rigaku Geiger Flex equipment using Cu, Kα radiation with 2θ varying from 5 – 90^{o}C. All the samples used were obtained from injection moulded tensile specimens. The results showed the presence of β phase through DSC and X-ray diffraction analysis of the nanocomposite, which is the result of the nucleation effect of $CaCO_3$ nano particles in polypropylene crystallisation process. The non-isothermal analysis showed that the melting temperature is not affected by $CaCO_3$ nano particles but the crystallisation temperature and crystallinity degree increase with the addition of $CaCO_3$ content.

Isothermal analysis showed that the incorporation of $CaCO_3$ nano particles reduced the half crystallisation time and increased the kinetic constant (k) which meant that the nano composites crystallized faster than the neat polypropylene due to the nucleation effect of the nano particles. Optical microscopy results showed a reduction in the spherullites size with incorporation of nano particles. From the results of their work, it is clear that $CaCO_3$ nano particles affected the crystallisation process of Polypropylene by changing its phase formation, crystallisation temperature, spherullites morphology and kinetics. Strapasson *et al.,* (2005) reported that the addition of polypropylene to polyethylene, gave a significant decrease in impact strength, with partial sample fracture for the 25%LDPE content blend. Further PP addition made the blend

behaviuor change from ductile to brittle and their blends were processed in an injection-moulding machine with various PP/LDPE. The linear law of mixtures was obeyed for all blends regarding elastic modulus and yield strength, except those in which polymer degradation was the determinant factor. Elongation at break, however, demonstrated incompatibility for this blend, unambiguously seen for the 50% PP content blend, which showed the lowest elongation at break of all compositions.

Hanim *et al.,* (2008) studied the effects of calcium carbonate nano-filler on the mechanical properties and crystallisation behavior of polypropylene. In their study, PP/$CaCO_3$ nanocomposites were prepared using a co-rotating twinscrew extruder at filler loadings of 5, 10, and 15% weight. The mechanical properties of the nanocomposites were evaluated using impact, flexural and tensile test, while the crystallisation behavior was analyzed using DSC and WAXD techniques. The impact strength and Modulus of polypropylene showed some improvement with the incorporation of nano filler while the tensile strength deteriorated. Scanning electron microscope photomicrographs showed evidence of calcium carbonate agglomeration within the polypropylene matrix, indicating that the level of shear stress generated during melt compounding was far from adequate to break-up the $CaCO_3$ nano filler. WAXD results showed the appearance of β-phase polypropylene in the nanocomposites with 10 and 15-weight percentage $CaCO_3$. The influence of $CaCO_3$ nano filler on the crystallisation behaviour of Polypropylene were also investigated using a DSC. Incorporation of $CaCO_3$ shifted the crystallization isotherms of polypropylene towards higher temperatures, indicating that the nano filler has acted as a nucleating agent for polypropylene. The reduced values of half crystallization times also implied that the introduction of $CaCO_3$ has accelerated the

crystallization rate of polypropylene. In their work, stearic acid coated calcium carbonate nano-filler were used to ensure uniform dispersion in the polypropylene matrix.

Xie *et al.,* (2004) successfully synthesized PVC/$CaCO_3$ nanocomposites via in-situ polymerization technique with filler loadings of 2.5, 5.0 and 7.5 weight percents. Their studies revealed that the toughness and stiffness of PVC were simultaneously improved with the addition of 44nm sized $CaCO_3$ filler. Uniform filler dispersion was obtained using the technique employed and they concluded that the toughness effect observed was attributed to the debonding/voiding of the nano $CaCO_3$ particles and the deformation of the matrix material surrounding the nanoparticles.

Di Lorenzo *et al.,* (2002) successfully polymerized poly ethylene terephthalate (PET) with coated and uncoated $CaCO_3$ particles of 40 – 80 nm size. They found that stearic acid coated $CaCO_3$ resulted in a stronger polymer-filler interaction compared to the uncoated grade. The strong interfacial adhesion between the phases resulted in significant increase in the glass transition temperature (14^oC shift) and melting temperature (8^oC shift) of the nanocomposites.

Chan *et al.,* (2002) prepared PP/ $CaCO_3$ nanocomposites by melt mixing in a Haake mixer using various mixing time (15, 30 and 45 minutes) with filler loadings of 4.8, 9.2 and 13.2 volume percents. The dispersion of Calcium carbonate nanoparticles in PP was good for filler content below 9.2% volume. Their Differential scanning calorimetry results indicated that the nano $CaCO_3$ are very effective nucleating agents for polypropylene. Tensile tests showed that the modulus of the nanocomposites increased by approximately 85%,while the ultimate stress and strain as well as yield stress and strain were not much affected by the presence of $CaCO_3$ nanoparticles. The results of the tensile test can be explained by the presence of the two-counter balancing forces: the reinforcing effect of the $CaCO_3$ nanoparticles and the decrease in the

spherullite size of the polypropylene. Izod impact tests suggested that the incorporation of $CaCO_3$ nanoparticles in PP significantly increased its impact strength by approximately 300%. J-integral tests showed a dramatic 500% increase in the notched fracture toughness. Micrographs of scanning electron microscopy revealed the absence of spherullitic structure of PP matrix. In addition; DSC results indicated the presence of a small amount of β-phase PP after the addition of the $CaCO_3$ nanoparticles.

Ustarroz *et al.,* (2005) reported that the Carbon/PP and carbon/self-reinforced PP hybrid composites have been produced by film-stacking. Tensile tests in carbon/PP composites show that the Elongation at break -modulus was 15% lower than expected. Compared to carbon/PP materials, hybrid composites showed an increase of 6-20% in ultimate strain and 10-15% in strength, while at the elongation at break-modulus was kept as predicted.

Sihama *et al.,* (2013) revealed that the comparative studies on the mechanical properties of high density polyethylene/polypropylene (hdpe/pp) and low density polyethylene/ polypropylene (ldpe/pp) binary blend. Morphological analysis using scanning electron microscope, Preparation of Samples by melt mixing in an extruder, Mechanical Tests, Hardness Test, Impact Test and Creep Test. Binary Blends (HDPE: PP) gave higher values of tensile strength, fracture strength, young's modulus, hardness, creep rate and creep modulus than LDPE : PP with the blend of ratio 20% HDPE : 80%PP showing superior Mechanical properties.

Gui *et al.,* (2007) studied the comparison between two types of polypropylene (PP) with different molecular structure, namely, homogeneous polypropylene (HPP) and polypropylene block copolymer (PPC) blended with low density polyethylene and found that the mechanical properties of the LDPE/PPH blend were much higher than that of LDPE/PPC blend, which was

attributable mainly to the fact that the mechanical properties of the neat HPP were stronger than that of the neat PPC.

Also Dikabe and Luyt, (2010) studied the morphologies as well as the mechanical properties of two types of blends, polypropylene/linear low density polyethylene and maleic anhydride grafted polypropylene MAPP: LLDPE, PP/LLDPE blends. They showed better properties and thermally stable than the (PP/LLDPE) as a result of stronger interaction between MAPP and LLDPE as compared with PP and LLDPE.

2.2 Components of Composite Material

2.2.1 Matrix

2.2.1.1 Polypropylene

Polypropylene [$–CH_2 – CH –CH_3–$]n is a strong and light weight thermoplastic that offers outstanding toughness, rigidity, and resistance to thermal deformation. Fabricators and designers value these characteristics and have considered polypropylene as one of the most satisfactory thermoplastic resins for a wide range of applications. This unique material can be steam-sterilized or autoclaved without damage and resists environmental stress-cracking when subjected to most chemical tests. Several characteristics of polypropylene enable thin section oriented mouldings to have virtually unlimited flex life, making it an excellent material for integral hinges in moulded parts.

2.2.2 Polymerization

The monomer of PP is obtained from the cracking of petroleum and as a by-product in oil refineries. Isotactic polypropylene can be obtained by the polymerization of propylene in the

presence of Ziegler-Natta catalyst (aluminium trialkyl and titanium tetrachloride) (Ebewele, 2000). Factors affecting the processing of polypropylene include injection moulding, cylinder, temperature, injection pressure, injection time, mould temperature, curing time, back pressure, screw speed, mould release, interface characterisation, micromechanical technique, finishing hot stamp printing, vacuum metalising, surface treatment, gas flame, corona discharge (electrical discharge), machining properties, turning, chasing, sawing, drilling and tapping, milling and shaping, joining and welding methods, spin welding, hot gas welding, hot plate welding or butt welding, heated tool welding, thermal impulse sealing, ultrasonic welding, effects of ultraviolet radiation (UV), microwave, effect of *gamma* radiation on PP and regrind,

2.2.3 Properties

Polypropylene can be obtained in isotactic, syndiotactic and atactic forms. Isotactic polypropylene is highly crystalline and with a crystalline melting point of 176^{o}C and density of 0.92g/cm^{3}. The mechanical properties of polypropylene depend upon the degree of crystallinity, molecular weight and molecular weight distribution. High crystalinity imparts a high tensile strength, stiffness and hardness to isotactic polypropylene. It has excellent electrical properties, inertness to chemicals and moisture resistance. However, at low temperatures, its impact strength is impaired.It is inherently less stable than polyethelene to heat, light, and antioxidants' attack (presumably because of the presence of tertiary hydrogen). It has low resistance organic solvent attack; resistance is below 80^{o}C and susceptible to thermooxidative degradation. These limitations must be minimized and/or even eliminated, thus, additives such as fillers, antioxidants and ultraviolet absorbers must be added for satisfactory processing and weathering, (Billmeyer, 2005). Polypropylene is highly responsive to injection speed and pressure and set up quickly in the mould, enabling moulders to attain high production rates. This combination of

performance properties gives polypropylene a position in the injection moulding field that is unique among thermoplastics. Polypropylene also demonstrates excellent chemical resistance, good abrasion resistance, good dimensional stability, and a high surface gloss on finished pieces. The versatility of this polymer makes it particularly well suited for film and fibres requiring superior strength, optical qualities, grease resistance, and moisture barrier properties.

It is also cheaper than polyethylene and available in large quantities from the high temperature cracking of petroleum hydrocarbon and propane. Liquid or semi liquid polymers of propylene have been obtained by the use of such catalysts as phosphoric acid and amorphous structures having little use or importance. The use of Zeigler and supported chromium oxide catalysts has led, however, to the production of highly crystalline and largely isotactic propylene, which softens at about 160^{o}C, compared to the range 105^{o}C to 130^{o}C for polyethylene and has a tensile strength said to exceed that of 6-6 nylon. Products made from such polypropylene are harder, stiffer and more resistant to scratching than are those made from polyethylenes. Large-scale production of such polypropylene is now being carried out in many countries. In addition to use for moulded articles it is also used for coatings and its high crystallinity and tensile strength makes it a useful fibre-forming polymer.

2.2.4 Applications

It can be injection-moulded into auto-motive parts. Another important use is in the manufacture of filaments for bagging crops, etc. and suitable for carpeting, polypropylene compares favourably with nylon in its fibre properties.

2.3 Reinforcements functions on polymer composite

Fillers are particulate materials added to polymers in order to improve physical properties of the polymer and to reduce cost of the composites. They can be classified according to their source function, composition, and/or morphology. No single classification scheme is entirely adequate because of the overlap and ambiguity of these categories. The chemical composition and its effect on composite physical properties, typically classifies fillers into three broad categories nonreinforcing or degrading, semi reinforcing or extending, and reinforcing fillers. In other words, Fillers: are inert foreign substances added to a polymer to improve or modify its properties. Fillers are more often added to plastics to improve tensile and compression strength, abrasion resistance, toughness, dimensional stability, and other properties. Materials used as particulate fillers include wood flour (finely powdered saw dust), silica flour and sand, glass, clay, talc, limestone, and even some synthetic polymers. Particles size range from 10nm to microscopic dimensions. Polymers that contain fillers can be classified as composites materials. Often, the fillers are inexpensive materials that replace some volume of the more expensive polymers,thereby reducing the cost of the final product.

Use of fillers in many commercial polymers is for the enhancement in stiffness, heat distortion temperature, damping impermeability and cost reduction, although, not all of these desirable features are found in any single filled polymer. Improvements in composite physical properties are directly related to the particle size, where the smaller particulate fillers impart greater reinforcement. Particle size distribution and particle shape also have significant effects on composite reinforcement. Filler structure ranges from precise geometrical forms, such as spheres, hexagonal plates, or short fibres to irregular masses. A particle with high aspect ratio has higher reinforcement than a more spherical one. Fillers having a broad particle-size distribution have

better packing in the polymer matrix and provide lower viscosity than that provided by an equal volume of the filler with a narrow particle size distribution. The properties of the particulate-filled polymers are determined by the properties of the components, by the shape of the filler phase, by the morphology of the system, and by the polymer-fillers interfacial interactions. Traditionally, most fillers were considered as additives which, because of their unfavourable features, surface area, or chemical composition, could only be moderately increase the modulus of the polymer, whereas strength (tensile, flexural) remained unchanged. Their major contribution was in lowering the cost of materials by replacing the most expensive polymer; other possible economic advantages were faster moulding cycles as a results of increase in thermal conductivity fewer rejected parts due to warpage. Additional functions may include degradability enhancement, barrier characteristics, antiaging characteristics, bioactivity, radiation absorption, warpage minimisation and so on, (Mascia, 1974).

Depending on the type of filler, other polymer properties could be affected, for example melt viscosity could be significantly increased through the incorporation of fibrous materials. On the other hand, mould shrinkage and thermal expansion would be reduced, a common effect of most inorganic fillers (Malcrum *et al.*, 1997). Examples of fillers include: wood flour, Asbestos graphite, mica, jute fibre, calcium carbonate, cotton and other textile fibrics.

2.3.1 Classification and types of fillers

The fillers can be divided into two classes; the organic type fillers e.g. cellulosics and the in-organic type fillers e.g. Asbestos.

The properties sought in fillers are; low cost and abundant supply, compatibility and ease of mixing with resin and other additives, high mechanical strength, low moisture absorption, high

heat resistance, good electrical conductance, ease of moulding and absence of abrasive or chemical reaction on mould.

The term filler is very broad and encompasses a very wide range of materials. They may be of variety of solid particulate materials (inorganic, organic that may be irregular, a circular, fibrous or plate-like in shape and are used in reasonably large volume loading in plastics. Pigments and elastomeric materials are normally not included in this definition. There is a significant diversity in chemical structure, form, shape, size, and inherent properties of the various inorganic and organic compounds that are used as fillers. They are rigid materials immiscible with the matrix in both molten and solid states, and as such, form distinct dispersed morphologies. Wypych, (2000) reported that there are more than 70 types of particulates or flakes and more than 15 types of fibres of natural or synthetic origin that have been used or evaluated as fillers in thermoplastics and thermosets.

A more convenient scheme, first proposed by Mascia for plastic additives, is to classify fillers according to their specific functions, such as their ability to modify mechanical, electrical, or thermal properties, flame retardency processing characteristics, solvent permeability, or simply formulation costs. Fillers, however, are multifunctional and may be characterised by a primary function and a plethora of additional functions. The scheme adopted involves classification of fillers according to five primary functions, as follows: mechanical property modifiers (and further subdivision according to aspect ratio), fire retardants, electrical and magnetic property modifiers, surface property modifiers and processing aids.

2.3.2 Types of fillers

Wood flour:

The most widely used filler is wood flour, particularly for formaldehyde resins. For this purpose, light coloured woods such as pine and spruse are the preferred ones although hard woods of maple, birch and oak have also been used. The material used is first separated from knots and back. The moisture content is adjusted to 2-3% and finally the wood is ground to the required mesh size. The latter process is of considerable importance. The process used is called attrition grinding. Although, the wood is ground to a fine mesh size, the fibres are left in an undamaged condition.

Wood flour has low specific gravity in extending the resin due to the greater volume of moulded products formed per kilogram of the base. Its general characteristics also favours its wide spread use. Some of these are its excellent mould ability, good shock resistance, high tensile strength, desirable electrical machine. However, there are draw backs as well: the moisture resistance is poor and it suffers from shrinkage in service. The temperature resistance is not high enough ($135^{o}C$), which restricts its use. When pastel shades are required, for example in urea formaldehyde resins, a special grade of cellulose known as alpha cellulose is used in place of wood flour (Callister Jr, 2010).

Cotton and other textile fabrics:

Where high strength is required, cotton flocks or finely ground cotton is generally used. Use of cotton flocks imparts high impact and tensile strength to molded objects. Fabric scraps, clippings and rags are bleached, purified and shredded and then used as fillers. However, there are certain limitations to the use of fabric fillers. The moisture absorption of moulded pieces is high and

merchantability is more difficult because of the exposure of fabrics at the surface. Further, the mouldability is decreased because of high bulk ratio of the moulding powder.

Asbestos:

Asbestos is mineral filler which is the hydrated form of magnesium silicate. In its natural form, it consists of small fibre admixed with some impurities. This leads to some loss of strength in its action as a filler compared to cotton fabrics, but this is compensated for by the improvement in other properties. The moisture absorption of moulding is very low and its electrical characteristics are excellent. The plastics employing asbestos as filler such as asbestos filled phenolics have excellent heat resistance and can withstand temperatures up to 200^{o}C. The mouldings are also resistant to chemicals.

Graphite

Graphite is used as filler to a limited extent because of its lubricating value. The lubricating influence is evident during the moulding operation. It can be very easily removed from the mould. Due to this property, it finds use in self-lubricating bearings.

Mica

One grade of mica in plastics is a finely wet-ground, white viscovite. Its electrical characteristics and high heat resistance are important. The advantages in using mica are that it has prominent electrical characteristics and high heat resistance. It has low relative density of 226. It has no abrasive effect on the mould, it is easily wetted by resins and dyes and does not absorb moisture, its light colour is retained at elevated temperatures. The main drawback is that mica has a tendency to cause peeling or sticking to the mould as phenol does not coat the mica sufficiently.

Jute fibre

Jute fibre, yarn and fabrics have been used for reinforcement in phenolic compositions. These fabrics have low strength but this deficiency is overcome in part by using parallel jute fibre.

Calcium carbonate:

Chemical identity:-

Chemical name: Calcium carbonate

IUPAC name: Calcium trioxocarbonate (IV)

Common name: limestone

Molecular Formula: $CaCO_3$

Molecular weight: 100 g/mol

2.3.3 Physical properties, uses and health effects

Calcium carbonate is a white powder. It has limited water solubility and has no flammable, explosive or oxidizing properties. Calcium carbonate solutions in water are slightly basic (pH>7). The pH of a saturated solution in water is 9.4. Calcium carbonate ($CaCO_3$) is a fine white powder. It is used in a broad range of industrial, professional, and consumer applications. Calcium carbonate does not have any toxic effect to man and the environment. Calcium carbonate is manufactured from calcium oxide. First, calcium oxide is allowed to react with water to form calcium hydroxide. Subsequently, the calcium carbonate is precipitated with carbon dioxide. Calcium carbonate is used as such or in a mixture for the production of articles to be used in or for vehicles, construction, electronic apparatus, laboratories, fabrics, wood, rubber, plastics, metal, leather, chemicals, pharmaceuticals, pesticides, cosmetics, personal care products etc. After entering the body, calcium carbonate will dissociate into calcium and carbonate ions. Calcium is an essential mineral in human nutrition. It serves as structural element

in bone, is involved in several nervous signaling systems and plays an important role in muscle functions. In the stomach, carbonate ions will react and carbon dioxide will be formed. When exposed via other routes, the carbonate will be absorbed and added to the already present pool of these ions in the body. The substance is not an irritant to the skin, the eye, and the respiratory tract. Calcium carbonate has no toxicity after single and after repeated oral exposure for prolonged periods. The substance is not a skin sensitizer, does not cause genetic effects or cancer and has no effect on male or female fertility, conception, and foetal development.

2.3.4 Environmental impact

Due to its limited solubility, calcium carbonate precipitates and deposits on the sediment, Calcium carbonate is a constituent of natural soils. Dissolved calcium carbonate dissociates into calcium and carbonate ions. Calcium ions will be assimilated by living organisms in the water and the carbonate will become part of the carbon cycle. Calcium carbonate is not toxic for the environment, (safety data sheet, 2011).

2.3.5 Classification of $CaCO_3$

Calcium Carbonate ($CaCO_3$) can be classified as:-

Mineral ground or Natural

Precipitated or Synthetic

Naturally occurring $CaCO_3$ is found as chalk, limestone and marble.

A typical composition of commercial grade $CaCO_3$ is shown below:

$CaCO_3$: 98.5 – 99.5%

$MgCO_3$: up to 0.5%

Fe_2O_3: up to 0.2%

(Material safety data sheet, 2011)

Other impurities include Silica, Alumina and Aluminum Silicate, depending on the location and source of the ore.

Loadings of $CaCO_3$ in PP typically run from 10 to 50%, although concentration as high as 80% has been produced (Karger-Kosis, 1995).

2.3.6 Uses of $CaCO_3$ as filler

$CaCO_3$ is usually selected as filler when a moderate increase in stiffness is desired.

It also increases the density of the PP compound; reduces shrinkage, which can be helpful in terms of part distortion and the ability to mould in tools designed for other polymers. At typical levels of 10 to 50%, the $CaCO_3$ does not significantly affect the viscosity of the compound.

It helps to disperse the finer-particle size $CaCO_3$. It also helps to prevent the absorption of stabilizers into the filler. Finally, as an added benefit, it acts to cushion the system, resulting in improved impact. The dispersion qualities of $CaCO_3$ particles play a crucial role in its toughening efficiency (Mgbemena, 2010).

2.3.7 Cashew tree

Cashew is a small tree with a usually small and crooked trunk. The leaves are simple, smooth, alternate, ovate 10 to 20 centimetres long 7 to 12 centimeters wide, with slightly rounded imaginative apex. The flowers are small 5 to 6 millimetres in diameters, crowded at the tips of the branches and yellow to yellowish white, the petals usually with pink stripes. The fruit, a nut, is as in coloured, kidney-shaped and about 2 centimeters long. The mesocarp is soft, corky and oleoresinous and the apicarp is leathery. Seed is fleshy, juicy, yellowish pear shaped and 5 to 7 centimeters long.

2.3.8 Distribution

Throughout the Philippines in settled areas at low medium altitudes, cultivated and in some places naturalized or at least persistent after being abandoned introduced from tropical America in the early colonial period called pantropic

2.3.9 Constituents cashew

Karnel yield or fixed oil, 45-47.2% starch; sitosterin, 8% cardonol; anacardic acid, lignoceric acid, oil contains linoleic acid, 7.7%, palmitic acid, 6.4%; stearic acid, 11.24%; lignoceric acid; 0.5% and sitosteric. The plant yields two oils: A light yellow oil from the pressed kaernels of which the finest quality is comparable to almond oil cardole from the shell of the nut, and acrid and powerful fluid useful for preserving cerved wood, books etc. against white ants.

Cashew nut oil yields: 16.12% moisture 2.3% ash, 3.94%; protein, 31.67% nitrogen, 5.70% crude fibre, 0.44% and carbohydrates, 45.46%. FAO, (2011)

The bark yields a gum also obnoxious to insects Kernel contains 7.6-16% moisture, 18-24% protein, 43 – 57% fats, 19-21% carbohydrates.

Nigeria was the world's largest producer of cashew nuts with shell in 2010.

Cashew nut production trends have varied over decades. African countries used to be the major producers before 1980s; India became the largest producer in the 1990s, followed by Vietnam which became the largest producer in the mid-2003. Since 2008, Nigeria has become the largest producer (Food and Agriculture Organisation, 2011).

Anacardium, the byproduct of cashew processing, has medicinal use and study of aqueous and methanol extract of leaf back and root of cashew yielded bioactive principles, that is tannin-15.38mg/g, total polyphenolics-2.00, alkaloids – 39.90 and oxalate -8.3, (Gisel *et al.*, 2012).

2.3.10 Polymers and polymer composites

Almost 85% of the polymers produced worldwide are thermoplastics, (Xanthos *et al.*, 1996). They can be divided in two broad classes, amorphous and crystalline depending on their characteristic transition temperatures, glass transition temperatures T_g, a temperature above which the modulus rapidly decreases and the polymer exhibits liquid-like properties; amorphous thermoplastics are normally processed at a temperature may be as low as 65^{o}C for polyamideimide (PAI) and as high as 295^{o}C for ploy amide (PA). Crystalline thermoplastics or more correctly, semi-crystalline can have different degrees of crystallinity ranging from 20 to 90%, they are normally processed above the melting temperature Tm of the crystalline phase and the Tg of the coexisting amorphous phase. Melting temperature can be as high as 365^{o}C for polyetherketone (PEK), as low as 110^{o}C for low- density polyethelyne (LDPE), and even lower for ethylene vinyl acctate (EVA) copolymers. Upon cooling, crystallization must occur quickly, preferably within a few seconds. Additional crystallization often takes place after cooling and during the first few hours following melt processing. Over 70% of the total production of the thermoplastics is accounted for by the large volume, low-cost commodity resins polyethylene (PE) of different densities, isotactic polypropylene (PP), polystyrene (PS), and poly (vinyl chloride) (PVC). Next in performance and cost are acrylics, Acrylonitrile-butadiene-stryrene (ABS) terpolymers, and high- impact polystyrene (HIPS). Engineering plastics such as acetals, polyamides, polycarbonate, polyesters, polyphenylene oxides and blends thereof are increasingly used in high performance applications. Specialty polymers such as liquid-crystal polymers polysulphones, polyamides, polyphenylene sulphide, polyetherketones, and flouropolymers are well established in advanced technology areas because of their high glass transition temperatures, $T_{g,}$, or melting temperatures, T_m (290-350^{0}C).

2.3.11 Polymer composites modification

Modification of organic polymers through the incorporation of additives yields with few exceptions, multiphase systems containing the additives embedded in a continuous polymeric matrix. The resulting mixtures are characterised by unique microstructures or macrostructures that are responsible for their properties. The primary reasons for using additives are; property modification or enhancement, overall cost reduction and improving and controlling of processing characteristics.

2.3.12 Types and components of polymer composites

Polymer composites are mixtures of polymers with organic or inorganic additives having certain geometrics (fibres, flakes and particulates). Thus, they consist of two or more components and two or more phases. The additives may be continuous, for example long fibres or ribbons; these are embedded in the polymer in regular geometric arrangements that extend throughout the dimensions of the product. Familiar examples are the well-known fibre-based, the most laminates that are usually classified as high performance polymer composites or as macro-composites based on the length of the fibres or ribbons. On the other hand, additives may be discontinuous (short), for example short fibre (say < 3cm in length), flakes, platelets or irregular (millimeters to micrometer size); fiber and flakes are usually dispersed in different orientation and multiple geometric patterns throughout the contentious matrix forming the micro composite.

Fillers intended for use in orthopaedic, bone regeneration, or tissue engineering application.

Additives for polymer composites have been classified as reinforcements, fillers, or reinforcing fillers. Reinforcements being much stiffer and stronger than the polymers usually increase its modulus and strength. Thus, mechanical property modifications may be considered as their

primary function, although their presence may significantly affect thermal expansion, transparency, thermal stability and so on (Raghupathi, 1990).

Composites may also be classified based on origin (natural versus synthetic) of the matrix or filler. Nature uses composites for all her hard materials. These are complex structures consisting of continuous or discontinuous fibrous or particulate materials embedded in an organic matrix acting as glue. Wood is a composite of fibrous cellulose and lignin. Bone is a composite of collagen and other proteins and calcium-phosphate salts. Spider silk consists of organic nanocrystals in an organic amorphous matrix. The shells of mollusks are made of layers of hard mineral separated by a protein bind. Such systems are usually based on thermoplastic matrix and are classified as lower performance polymer composites compared to their counterparts with continuous additives. When the fibres, platelets, or spheres as the dispersed phase are of nanoscale dimensions for example, hydrotalcite nanoplatelets, the materials are known as nanocomposites. They differ from microcomposites in that they contain a significant number of interfaces available for interactions between the intermixed phases, (Ajayan *et al.*, 2003).

2.3.13 Parameters affecting properties of composites

In general, parameters affecting properties of polymer composites include; properties of the additives (inherent properties, size, shapes), the composition of composites, the interaction of components at the boundaries, which is also associated with the presence of thick interface which is also known as the interphase; this is often considered as a separate phase, controlling the adhesion between the components and the method of fabrication.

2.3.14 Applications, trends, and challenges of fillers

Global demand for fillers for the plastics industry has been estimated to be about 15 million tonnes, (Mahajan, 2003). Primary end-use markets are building or construction and transportation, followed by appliances and consumer products; furniture, industrial or machinery, electrical or electronics, and packaging comprise a small market segment. Flexural modulus and heat resistance are the two critical properties of plastics that are enhanced by the inclusion of performance minerals. Automotive exterior parts, construction materials, outdoor furniture, and appliance components are examples of applications benefitting from enhanced flexural modulus. Automotive interior and under hood parts, electrical connectors, and microwave containers are examples of applications requiring high-temperature resistance. Environmental acceptance and improved sustainability of automotive parts are attributes of composites containing natural fibres (Joshi *et al.*, 2004).

There are a significant number of technological advances that will undoubtedly contribute to the additional growth in the usage of certain functional fillers, For example wood-filled plastics, introduction of specially configured counter-rotating twin-screw extruders with vent zones to remove moisture (Wood, 2007).

Recent statistics (2007) estimates the U.S demand for fillers and extender minerals to a total of 3.2 million tonnes per annum, (Blum, 2008). Annual growth rates were estimated to be 2-3% with much higher rates for fire retardant fillers.

Some new exciting application areas for composites containing certain functional fillers are; structural materials with improved mechanical, thermal and barrier properties, electrical conductivity, and flame retardency, high-performance materials with improved UV absorption and scratch resistance, barrier packaging for reduced oxygen degradation, multifunctional fillers

that could release in a controlled manner corrosion sensing additives, corrosion inhibitors, insecticides, active pharmaceutical ingredient, and so on bioactive materials for tissue engineering applications, (Ha *et al.*, 2009).

2.3.15 Compounding and mixing processes

Mixing and compounding are terms used interchangeably by different authours. Compounding is a major processing operation in fabricating plastics and their products. It is essentially simple mixing in which particles of two or more components are rearranged into a more random distribution without reducing the ultimate particle size (Mascia, 1974).

Its principle involves heating the blend in a closed mould whereby the resin softens and takes the shape of the mould. Temperature, pressure and time will depend on the nature of the material, size and shape of the mould, Holmes-Walker (1975).

The purpose of mixing in polymer processing is to attain an acceptable degree of homogeneity or uniformity of composition, (Holmes-Walker, 1975). According to reports, optimum results in mixing can be obtained when the mixing parameters, for example, time of mixing, temperature and speed are taken into account during speed mixing (Titow, 1984). The expected degree of mixing is in most cases identified by visual appearance of the mix and the homogeneity of the product after subsequent processing. Several types of machines such as roll mills, high-speed mixers, extruders and others are used in mixing. Each method of mixing imposes its own pattern and degree of severity on the matrix materials and additives. The conventional two roll mills was used in this studies for compounding the composites.

2.4 Plastic Processing Additive

2.4.1 Plasticizers

These are the additives which reduce the intermolecular forces between polymer chains thus act as internal lubricant. (Misra, 2008).Flexibility, ductility, and toughness of polymers may improve with the aid of plasticizers. Their presence also produces reductions in hardness and stiffness. Plasticizers are general liquids with low vapour pressure and low molecular weight. The small plasticizer molecule occupy the position between the large polymer chains, effectively increasing the inter chain distance with a reduction in the secondary intermolecular bonding. Plasticizers are commonly used in polymers that are intrinsically brittle at room temperature. The plasticizers lower the glass transition-temperature, so that at ambient conditions they may be used in applications such as thin sheets or films, tubings, raincoats and curtains.

2.4.2 Stabilizers

Some polymeric materials under normal environmental conditions are subject to rapid deterioration in terms of mechanical integrity. The additives that counteract deteriorative processes are called stabilizers. One common deterioration that result from exposure to light, particularly ultraviolet (UV) radiation, is that ultraviolet radiations interacts with the polymer and causes severance of some of the covalent bonds along the molecular weight chains, which may also result in cross linking. There are two primary approaches to UV stabilization. The first is to add UV absorbent material, often as a thin layer at the surface. This essentially acts as a sunscreen to block out the UV radiation before it can penetrate into and damage the polymer. Another important type of deterioration is oxidation. It is a consequence of the chemical interaction between oxygen as either diatomic (O_2) or ozone (O_3) and the polymer molecules.

Stabilizers that protect the polymer against oxidation either consume oxygen before it reaches the polymer and/or they prevent the occurrence of oxidation reactions that would further damage the material.

2.4.3 Colourants

Colourants impart a specific colour to a polymer; they may be added in the form of dyes or pigments.

The molecules in a dye are actually dissolved in the polymer. The pigments are filler materials that do not dissolve, but remain as a separate phase. They have small particle size and a refractive index near that of the parent polymer.

2.4.4 Flame retardants

The flammability of polymeric materials is a major concern, especially in the manufacture of textiles and children's toys. Most polymers are flammable in their pure form; exceptions include those containing significant amounts of chlorine and/or fluorine such as poly(vinylchloride) and poly(vinyl fluoride). The flammability of the remaining combustible polymers may be enhanced by flame retardants. These flame retardants may function by interfering with the combustion process through the gas phase, or by initiating a different combustion reaction that can generate less heat, thereby reducing the temperature; this causes a slowing or cessation of burning (Callister Jr, 2010).

2.5 Thermoplastics processing techniques

The processing of thermoplastic polymers is important to plastics scientists, engineers, and technologists. There are a number of processing methods, the principal ones are: extrusion, injection moulding, blow moulding and calendaring.

2.5.1 Extrusion

In the extrusion process, polymer is propelled continuously along a screw through regions of high temperature and pressure where it is melted and compacted, and finally forced though a die shaped in to the final object. A wide variety of shapes can be made by extrusion, including rods, channels, tubing, hose, sheets and films of similar width down to a few thousandths of centimetres in thickness.

The screw of an extruder is divided in to several sections, each with a specific purpose (Chung, 1977). The feed section picks up and finally moves the polymer from a hopper and propels it into the main part of the extruder. In the compression section, the loosely packed feed is compacted, melted, and formed into a continuous stream of molten plastic. Some external heat must be applied, but much is generated by friction. The metering section contributes to uniform flow rate, required to produce uniform dimensions in the finished product, and builds up sufficient pressure in the polymer melt to force the plastic through the rest of the extruder and out of the die. Since viscous polymer melts can be mixed only by the application of shearing forces (their viscosity is too high to allow turbulence or diffusion to contribute appreciably to mixing), an additional working section may be needed before the die.

Modern trends in extruder usage include the twin-screw extruder, in which two screws turn side by side in opposite direction, providing more working of the melt, and the vented extruder, having an opening or vent at some points along the screw that can be opened or led to vacuum top extract volatiles form the polymer melt.

2.5.2 Types of extrusion

Screw extrusion

The screw extrusion method of forming plastic is probably the most important method in comparison with the other forms or methods. This is because most thermoplastics will pass through an extruder at least once in their processing history.

Basically, the screw extrusion method consists of melting the polymeric materials and forcing it through a die. The shape of the die determines the shape of the final product or object.

The finely divided polymer chips (either as granules or in powder form), is fed through a hopper and pushed into the main helical screw via the feed pocket. The polymer then passes through the three main sections of the feed, compression and metering. The feed section of the screw, as the name implies, feeds and compacts the polymer solids which then pass to the compression section where melting occurs. The metering section controls the output of the machine. From the screw, the molten polymer is formed through the perforated beaker plate into the die. Using the screw extrusion method of thermoplastics processing, a variety of shapes such as rods, tubing, hose sheets and films are obtained.

Co-extrusion

Films or sheets consisting of layers of two or more different polymers can be produced by mixing the molten streams from a like number of extruders in a multiunifold die. This process can be used to combine minerals to provide combinations of properties that cannot be obtained in a single polymer. For example, a film for packaging food may consist of three layers impacting, respectively, high strength, low oxygen permeability, and heat stability (Brown 1981).

Film extrusion

The extrusion of a film can be carried out either by casting as in sheet extrusion or by the blown-film process. In the lather a tubular die is used from which a hollow tube of product is extruded vertically upward toward a film tower. The tube is blown into a thin cylindrical film by air introduced through the die and trapped in the film bubble. At the top of the tower, the film is collapsed, and subsequently slit into a flat film.

Pultrusion

In this variation of extrusion, material is pulled rather than pushed through a die or mould. The process is used to produce continuous length of fiber-reinforced thermosetting resins. The reinforcing materials are continuously pulled through a bath of liquid resin that saturates each individual fibre. Excess resin is removed by a forming guide, and the mix is then pulled through a heated mould that shapes and cures it (Martin 1979, Anderson, 1981).

Other extrusion processes

Wire and cable extrusion are other forms of extrusion and they employ dies through which bare copper or aluminum wire is passed and it is coated with plastic insulation. Extrusion rates can be as high as 8000m/s.

2.5.3 Moulding

Moulding processes are those in which a finely divided plastic is forced by the application of heat and pressure to flow into fill and conform to the shape of cavity (mould). One of the oldest methods of polymer processing, moulding can be carried out in different ways:

2.5.4 Compression moulding

Compression moulding is the least expensive and simplest of all polymer-processing operations. In compression moulding, the polymer is put between stationary and movable members of a mould. The mould is closed, and heat and pressure are applied so that the material becomes plastic, flows to fill the mold, and becomes a homogeneous mass. The necessary pressure and temperature vary considerably depend upon the thermal and rheological properties of the polymer and for a typical compression-moulding material they may be between 150^0C and 1000-3000N/m^2. A slight excess of the materials is usually placed in the mould to insure it is being completely filled. The rest of the polymer is squeezed out between the mating surfaces of the mould in a thin, easily removed film known a flash.

2.5.5 Injection moulding

Most thermoplastic materials are moulded by the process of injection moulding. Here, the polymer is preheated in a cylindrical chamber to a temperature at which it will flow and then forced into a relatively cold, closed mould cavity by means of quite high pressures applied hydraulically, traditionally, through a plunger or ram, but today almost invariably by means of reciprocating screw that serves the dual purpose of providing the molten polymer mass and forcing it into the mould. The screw rotates to pick up the particulate polymer, compact and melt it, mix the melt, and deliver it to the entrance to the mould. The screw then moves forward to force a fixed volume of the molten polymer into the closed mould. The melt temperature may be considerably higher than in compression moulding, and pressures of hundreds to thousands of tones are common. After the polymer melt has solidified in the cool mould, the screw rotates and moves backward to prepare the change of polymer for the next cycle. Meanwhile the mould is opened and the moulded article is removed.

An outstanding feature of injection moulding is the speed with which the finished articles can be produced. Cycle times of 10-30 seconds are common, as are multi-cavity mould allowing the production of many parts per cycle. Articles weighing up to many kilograms can be produced.

2.5.6 Blow moulding

This operation can be carried out either on an extruder or a reciprocating screw injection moulding machine. By means of compressed air or stream, the plastic; is then blown into the configuration of the mould. This technique is used in the production of hollow articles such as bottles and similar articles. In the case of large articles, such as two-litres beverage bottles, the perison may previously have been injection moulded and oriented to provide additional strength to the final blow piece.

2.5.7 Reaction-injection moulding (RIM)

This is relatively a new method with the unreacted components that lead to a polymer product. They are pumped in predetermined amount into a mixing head, where they are thoroughly mixed and injected into a warm (65^0C) mould under relatively low pressures (50-100N/m^2). Polymerization takes place in the mould. Meanwhile, the mixing head is cleaned, with an unused monomer been recycled. To date, RIM has been used almost entirely for the moulding of polyurethanes, but commercialization of nylon and epoxy RIM is anticipated.

2.5.8 Rotational moulding

In this technique, powdered polymer is loaded into a relatively inexpensive closed mould, which is intensively heated while being rotated biaxially. The polymer coats the inner walls of the mould to a uniform thickness and is faced there. The method has advantages for producing large hollow parts, and can be used to produce multiwall constructions by successive steps.

2.5.9 Calendaring

Calendaring is a process used for the continuous manufacture of sheets of film. Granular resin or thin sheet is passed between pairs of highly polished heated rolls under high pressure.

For production of film a series of pairs is used with a gradual reduction in roll separation as the stock progresses through the unit. Proper calendaring requires precise control of roll temperature, time, pressure and speed of rotation. By maintaining a slight differential between a roll pair, it is often possible to impart an exceedingly high gloss to the film or sheet surface. An embossed design can be produced on the surface by means of a calendar roll, appropriately engraved. By calendaring a mixture of granular resin chips of varying colour, this technique is widely employed in the manufacture of flooring compositions.

Other processing techniques include casting, transfer moulding, twin-screw and multi-screw extruder (Sunmonu, 1994).

2.6 Characterisation of Composites

2.6.1 Mechanical properties of plastics

2.6.2 Hardness

Hardness, as applied to plastics and its products is the relative resistance of the surface of indentation or penetration by an indentor of specified dimension under a specified load. Numerical hardness values represent either depth of penetration or convenient arbitrary units derived from depth of penetration. Hardness is usually the first requirement to be met in developing a mix.

There are two devices for measuring hardness. The first is composed of the durometers. The harder the sample, the further it will push the indentor pin and the higher the numerical reading on the dial. The second class of durometer instrument is the table top units unlike the durometer the softer the sample, the greater the numerical values representing hardness. A major limitation inherent in hardness measurement is that the characteristics that is measured, surface indentation rarely bears any relation to the ability of a plastics product to function properly. However, the nature of plastics, fillers, the degree of dispersion of ingredients etc, may affect hardness property of the vulcanisate (Walker, 1970).

2.6.3 Abrasion resistance

This is the resistance of a plastic's composition to wearing away by contact with moving abrasive surface. It is measured under definite conditions of load and speed and is expressed as a comparison in percentage with a standard composition. Abrasion may give rise to localized deformation occasioned by high temperature. At the last point, chemical reaction such as oxidation can occur which speed up the rate of abrasion thus leading to corrosion wear instead of pure mechanical wear. Abrasion is measured by the loss in weight or volume of material. The methods and instruments associated with them do not give the same results. Thus, caution may be required in using the data from abrasion test to produce actual performance behavior (Richardson, 1971).

2.6.4 Compression set and flex fatigue resistance

This is the amount of residual displacement in a plastic after a distorting load has been removed, compression set is time and temperature dependent and it is affected by the affinity of the plastic

for the filler surface. Compression set is an aspect of permanent set. Other aspects of permanent sets are permanent set in tension and in shear (Nelson, 1974).

2.6.5 Flex fatigue resistance

This is the ability of a material to withstand fatigue resulting from repeated distortion by bending, extension or compression. There is also flex cracking which is the deformation or surface crack as a result of repeated bending or stretching of the specimen. There are many kinds of fatigue tests. These include tensile or flexural tests at constant deformation and constant stress. Fatigue is due to the progress growth of cracks which contain flaws. One or more of the cracks grow until it is no longer microscopic in size, at this point it may propagate rapidly and cause failure factors, which increase the strength of polymer and may also tend to increase fatigue life. Among them are increase in molecular weight and orientation of the molecules parallel to the applied stress (Nelson, 1974).

2.6.6 Tensile strength, elongation at break and modulus

Tensile strength is the force per unit of the original specimen across sectional area applied at the time of the rupture of specimen. Elongation is the extension between benchmarks produced by a tensile force applied to a specimen and is expressed as a percentage of the original distance between the marks and benchmarks produced by the applied force often referred to as tensile stress. It is the stress in mega pascals required to produce a certain elongation.

In plastics, modulus is stress at a certain strain and not a ratio nor a constant, merely the coordinates of a point on the stress-strain curve i.e. stress and strain in tension is not proportional unlike steel. In filled system, an increase in modulus over the unfilled material indicates

reinforcement and the magnitude of the modulus values gives the level of reinforcement (Blow, 1982).

Ultimate elongation is the elongation at the moment of rupture. Both tensile strength and elongation are derived from stress-strain measurement. Their importance is that contamination, degradation and adulteration can be detected by these measurements.

Also a high tensile strength is usually an indication of good tear and abrasion resistance. Tensile properties in vulcanisate can be affected by the nature of elastomers, level of filler, the degree of fillers dispersion, temperature and time of curing (Dibion, 1984).

2.6.7 Resilience

This is the ratio of a deformed elastomer specimen i.e. ratio of energy given up on recovery from deformation to the energy required to produce the deformation. In filled systems, fillers with the least effect on resilience are those that are least reinforcing. Resilience can be determined from a low speed stress-strain loop, by impact test or by free-vibration or forced vibration device (Hofmann, 1996).

2.7 Morphological Characterisation of Composites

2.7.1 Spectroscopic tests

Electron microscopy for chemical analysis/X-ray photoelectron spectroscopy, mass spectroscopy, X-ray diffraction studies, electron-induced vibration spectroscopy, and photoacoustic spectroscopy are successful in polymer surface and interfacial characterisation.

2.7.2 Microscopic techniques

Microscopic studies such as optical microscopy, scanning electron microscopy, transmission electron microscopy, and atomic force microscopy can be used to study the morphological changes on the surface and can predict the strength of mechanical bonding at the interface. The adhesive strength of fiber to various matrices can be determined by Atomic Force Microscopic studies.

2.7.3 Thermodynamic methods

The frequently used thermodynamic methods for characterisation in reinforced polymers are Differential Thermal Analysis, Differential Scanning Calorimetry wettability study, inverse gas chromatography measurement, zeta potential measurement, and so on. Contact angle measurements have been used to characterise the thermodynamic work of adhesion between solids and liquids and surface of solids (Cook, 1968).

2.7.4 X-ray diffraction (XRD)

The atomic planes of crystals cause an incident beam of X-rays to interfere with one another as they leave the crystal the phenomenon is called X-ray diffraction. X-ray diffraction analysis measures the average spacings between layers or rows of atoms, determine the orientation of a single crystal or grain, find the crystal structure of an unknown material and measure the size, shape and internal stress of small crystalline regions.

2.7.5 Basics of crystallography

A crystal consists of a periodic arrangement of the unit cell into a lattice. The unit cell can contain a single atom or atoms in a fixed arrangement. Crystals consist of planes of atoms that

are spaced at distance apart spacing. Seven crystal systems: cubic, tetrahedral, hexagonal, rhombohedral, orthorhombic, monoclinic and triclinic

2.7.6 Production of X-rays

X-rays are produced whenever high speed electrons collide with a metal target. Specimen preparation of powders: 0.1m < particle size < 40m peak broadening less diffractions occurring double sided tape. Smooth surface after polishing specimens should be thermal annealed to eliminate any surface deformation induced during polishing.

Basic features of typical; XRD experiment are: production, diffraction, detection and interpretation.

Detection of diffracted X-rays by a diffractometer consists of Circle of diffractometer focalization circle, detector photon counter, amplifier and a recorder

2.7.7 Bragg's law and diffraction

States how waves reveal the atomic structure of crystals

$n\lambda = 2d\sin\theta$, where n is an integer and λ is the wave length of the X-ray at which the analysis was performed and d is the distance between adjacent planes.Diffraction occurs only when Bragg's law is satisfied condition for constructive interference (X-ray 1 and 2) from planes with spacing d. Significance of peak shape in XRD are; peak position, peak width and peak intensity

2.7.8 Applications of XRD

XRD is a non-destructive technique, used to identify crystalline phases and orientation, determine the structural properties, lattice parameters (10-4Å), strain, grain size, expitaxy, phase composition, preferred orientation level order/disorder transformation, thermal expansion, to

measure thickness of thin films and multi-layers, determine atomic arrangement and detection limits: 3% in a two phase mixture; can be spatial resolution normally none-Phase identification

The most important uses of XRD are to obtain XRD patterns, measured spacing, obtain integrated intensities and compare data with known standard in joint committee power standard file (JCPDS), which are for random orientations (there are more than 50,000 JCPDS card of inorganic materials). XRD studied temperature, electric field, pressure and deformation (Mythili CV *et al.*, 2015).

X-ray analysis: X-ray analysis is widely used to study the internal arrangement of polymer molecules and the changes that occur on the Polymer structure due to heat effects and mechanical effects, Clark (1963) and Tager (1978).

According to Billmeyer (1973) and Edward (1973) X-ray analysis of crystalline polymers have revealed the pressure of both ordered and disordered regions which are manifested in the form of sharp and diffused features. Various x ray techniques are employed in the study of processed samples; these include wide-angle x-ray diffraction (WAXD), small-angle x-ray diffraction (SAXD) and small-angle x-ray scattering (SAXS) each covering different Bragg angle, (Clark (1961) and Carrega (1977). The crystallite size may be obtained from the measured width of the X-ray diffraction line.

The Scherrer formula was for the calculation of crystallite size (grain diameter) D, of the sample Srivastava, 2011

$$D = \frac{k\lambda}{\beta \mathrm{Cos}\theta} \quad \text{..... equation (1)}$$

Where, D is the crystallite size, K is a constant varies with crystallite shape but usually nearly equal to 0.94, λ is the wavelength of source radiation and β is full-width at half maximum (FWHM) of the peak, in radians and θ is the Bragg's angle The crystallite size was calculated using Scherrer formula for the prominent peaks in the diffraction pattern for both sample PP/$CaCO_3$.

The percentage crystallinity (X_C) = $\frac{I_c}{I_c + I_a}$ x 100 ……. equation (2)

The crystanillity index (I_x) = $\frac{I_c - I_a}{I_c}$ x 100 ……... equaton (3)

Where I_c and I_a are the intensities of the crystalline and the amorphous region (prominent peaks) respectively.

2.7.9 Scanning electron microscopy (SEM)

A microscope that produces an image by the use of an electron beam that scans the surface of a specimen; an image is produced by reflected electron beams. Examination of surface and micro-structural features at high magnification is possible using this instrument. The major components of a Scanning Electron Microscope Machine are: primary operational systems, vacuum, beam generation, beam manipulation, beam interaction, detection, signal processing, and display and record. These systems function together to determine the results and qualities of a micrograph such as magnification, resolution, depth of field, contrast, and brightness. A brief description of each system follows: Vacuum system. A vacuum is required when using an electron beam because electrons it quickly disperse or scatter due to collisions with other molecules. Electron beam generation system: This system is found at the top of the microscope column and it generates the "illuminating" beam of electrons known as the primary electron beam. Electron

beam manipulation system: This system consists of electromagnetic lenses and coils located in the microscope column and control the size, shape, and position of the electron beam on the specimen surface. Beam specimen interaction system; this system involves the interaction of the electron beam with the specimen and the types of signals that can be detected. Detection system; This system consists of several different detectors, each sensitive to different energy or particle emissions that occur on the sample chamber. Signal processing system; this system is an electronic system that processes the signal generated by the detection system and allows additional electronic manipulation of the image. Display and recording system; this system allows visualization of an electronic signal using a cathode ray tube and permits recording of the results using photographic or magnetic media. (Callister's jr, 2010).

2.7.10 Thermoforming/solid phase forming

An intensive development effort by machinery suppliers has resulted in new mechanical forming equipment. The use of this equipment is expected to accelerate polypropylene's use in packaging applications. The amount of polypropylene used in the fabrication of thin walled food containers could grow dramatically over the next three to five years. Properties conducive to polypropylene's selection for packaging applications are resistant to chemical attack and high temperature, good impact/rigidity balance, low density, good barrier properties, and non-toxic characteristics. Hot-fill and microwave oven containers, for example, are fabricated with polypropylene because of its superior heat resistance. The resin's long term favourable supply and competitive pricing will be added inducements to growth. The primary deterrent to widespread use of polypropylene for thermoforming is its higher and narrower forming temperature range. The temperature at which sheet begins to bubble is only 15-20^{o}C above the transition temperature at which it becomes formable. Successful forming, however, can be

realized with a minimum of sagging if the sheet is heated above the 300°C softening point, but below the 333°C melting point. Within this range, polypropylene orients, resulting in improved clarity, strength, and barrier qualities. Above the melting point, the resin does not orient and its low melt strength at that temperature leads to sagging, which makes thermoforming difficult. Below the softening point, the resin's stiffness has a similar adverse effect. Temperature control is a critical processing condition for forming polypropylene, and maintaining the desirable range is difficult because of the resin's relatively poor heat conductivity. Consequently, preheating is required to prepare the sheet stock for forming. The temperature during forming must also be controlled. If it is not, thick and thin sections will result that cannot be moulded with accuracy. Finally, at the end of the cycle, heat must be extracted before the part can be removed. Ideally, temperature cycling should be as fast as possible in order to attain a high production rate. Moulds for polypropylene are generally made of aluminum, which has high thermal conductivity. Channels are used for carrying liquid coolant to hasten temperature cycling. Although moulds are important to proper thermoforming, equipment innovations have been the principal stimulus in the renewed interest in forming polypropylene. Machines currently available illustrate the two techniques developed. Stretch forming is used for cylindrical and rectangular containers. Where thin-walled items and heavy, hollow shapes are required, pressure forming techniques are employed. Processing at a temperature just below the softening point is a common requirement. In pressure forming, a multistage oven heats the sheet, which is clamped over the moulds. A heated plug forces the material into the mould cavity, followed by a blast of cold air that forces the hot sheet against the cooled mould surface. Then, the part is released. Other machine developments involve alternate approaches to handling the sheet during processing. A shuttle-mould system where the female elements shuttle back and forth across the web after forming and

trimming the part is popular for moulding cup. Trimmed parts cool in the mould cavity before ejection. Rotary systems are also employed. Instead of carrying sheet through the system in a straight line, preheating, forming, and trimming stations are arranged in a circle. Earlier and more complete control and elimination of sagging problems in the forming stage are readily attainable.

2.8 Physical Method of Characterising Composites

2.8.1 Solvent Sorption Measurement: Solvent sorption is one of the most widely used techniques in characterising structural changes in polymers. According to Gray and Gilbert (1975), the rate of solvent absorbed at equilibrium all depend on the type of polymer and its thermal or shear history. Illers (1977) reported that in semi crystalline polymers, solvent sorption behaviour is directly proportional to the amount of crystallinity and the available free volume in a polymer. Measurements of the equilibrium sorption of vapours or of gases in miscible blends can, in principle, give information about the interaction energy parameter. The rate of sorption of liquid by a resin following Fick's law of diffusion (Vergaud, 1991).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 MATERIALS

The following materials were used

1. Polypropylene
2. Cashew Nutshell Powder
3. Calcium Carbonate

3.2 Apparatus Used

The following apparatus were used in the Experiments

1. Mould
2. Hand gloves
3. Mixing knife
4. Weighing balance (triple beam balance, model 2016 MB)
5. Stop watch
6. Specimen Bottles
7. Oven
8. Saw blade
9. Sam paper
10. Microsrew gauge

3.3 Equipment Used and their Sources

S/N	Equipment	Specification	Sources
1.	Two roll-mill	Reliable Rubber and Plastic Machine Company New York Jersey, U.S.A (model 5189)	Nigerian Institute of Leather and Science Technology (NILEST)
2.	Hydraulic press	Carver Inc., Wabah U.S.A (model 3851-0)	Nigerian Institute of Leather and Science Technology (NILEST)
3.	Hardness Tester	Show A hardness tester model number 5019	Nigerian Institute of Leather and Science Technology (NILEST)
4.	Tensometer	Monsator Tensometer model number 9875 type W	Department of Mechanical Engineering Faculty of Engineering Ahmadu Bello University, Zaria
5.	Scanning Electron microscope Machine	Phenom World	Department of Chemical Engineering Faculty of Engineering Ahmadu Bello University, Zaria
6.	X-diffraction machine (XRD)	Shimadzu 6000 X-ray diffractometer	National Steel Raw Materials and Exploration Agency No:18 Rabah Road, Malali Kaduna
7.	Charpy Impactor	Cat. Nr, 15J x 25J	Department of Material Science and Metallurgical Engineering Faculty of Engineering Ahmadu Bello University, Zaria

3.4 Preparation of Sample

The cashew nutshell was picked up from the unwanted parts (shell), dried and kept for filler preparation.

3.4.1 Filler Preparation

The Cashew Nutshell powder (CNSP) was collected from the processing point in Auchi Local Government Area, Edo State, Nigeria in September, 2014. The sample was packaged in a cleaned polyethene bag and transported to the laboratory. It was dried at ambient condition in the laboratory for five months. When the sample record to be dry it was grounded to powder with a mechanical grinder and sieved through a sieve of mesh size 420µm.The sieved sample were stored separately in plastic container and labeled appropriately.

3.4.2 Mixing of the Compound

The two roll mill was used, equipment was switched on and the PP processing temperature was set at 170^{o}C. The nip of the rollers were adjusted and material was poured on the nip, after total melting had been attained, the filler (CNSP) was incorporated and then cross-mixed until a homogeneous mixture was obtained and the compound was sheeted out for further processing. The compounding was done base on the formulation proposed by Mamza, (2011).

Table 3.1

S/No	Mass of Ingredients (g)	1	2	3	4	5	6	7	8	9
1	Polypropylene	100	90	80	70	60	50	40	30	20
2	Cashew Nutshell powder	0	10	20	30	40	50	60	70	80

Table 3.2

S/No	Mass of Ingredients(g)	1	2	3	4	5	6	7	8	9
1	Polypropylene	100	90	80	70	60	50	40	30	20
2	Calcium Carbonate	0	10	20	30	40	50	60	70	80

3.5 Determination of Mechanical Properties of the Prepared Polypropylene Composites

3.5.1 Determination of Tensile strength

The tensile strength test was performed using an advanced material test machine (Tensometer 9875 type W) in accordance with the ASTM D638 method.

The test piece for tensile test had dimension (100mm X 30mm) with a bench mark of 40mm. The samples were individually clamped horizontally at both ends with the Jaws of the tensometers and were stretched horizontally to failure and the results were recorded. (ASTM D638, 1983)

3.5.2 Determination of Hardness of the prepared composites

The hardness test was carried out according to ASTM method by placing the indentor on the plastics material and the hardness readings were manually recorded as the pointer of the hardness tester. (ASTM D785, 1983)

The variation in the shore A hardness values was determined using Durometer hardness tester of model identity DIN53505, EN180868 in accordance with ASTM D22401807619. Manufactured by MUVER NO: 5019.

The Moulded specimens starting from 0 g filler loading to 80 g filler loading for both $CaCO_3$ and CNSP samples were analysed by IPHD (International Plastic hardness Degree). For each

specimen, six different spots were indented and the averages of the hardness values were calculated in every case.

3.5.5 Determination of Impact Strength of the prepared samples

The impact test of this research was carried out in accordance with American Society for Testing Materials (ASTM) with a Charpy impact machine of model number 412 of capacity 15 J for polymer composites and 25 J for metallic composites at room temperature. A test piece of dimension 100mm × 4mm was cut and placed in the Impactor sample holder, and the impact hammer was released and the impact values were recorded as averages.

3.5.6 Determination of microstructure of the prepared composites byScanning Electron Microscopy

The specimens were prepared and manipulated as little as possible to the appropriate size and stable in the vacuum, and were electrically coated with a metallic conductor which possessed the characteristics resembling the natural metallic state. The morphology of the cross-section of the Polypropylene composites of different compositions with or without the filler incorporated in the polypropylene matrix was examined by the scanning electron microscope,(SEM) at magnification of 5000x obtained by the Department of Chemical Engineering, Ahmadu Bello University, Zaria. The prepared composites of composition of fillers' composition 60/40, 50/50, PP/CNSP, PP/$CaCO_3$ and unfilled PP specimens were taken for scanning electron microstructure tests in order to determine and characterise the micro-structural contents of the PP/CNSP and PP/$CaCO_3$ at their microstructural level. The processed samples were mounted in to the sample holder and results were shown on the display screen.

3.5.7 Determination of the crystallinity of the prepared composites by X-ray Diffraction

The dried sample of the Cashew Nutshell Powder was sieved using sieve of 420μm mesh size, the sieved sample was then packed in a polyethene bag and transported to the laboratory for the analysis after which was then loaded on to the machine and runned in the sample chamber of the of the Shimadzu X-ray diffraction machine and maximum voltage (40kv) and a maximum current (30mA) was applied to produce the X-rays to excite the sample for a preset time (10minutes) in this case. The spectrum obtained from the sample is now analysed to determine the constituent elements and possible compounds present in the cashew nutshell. The X-ray diffractometer used for this research was Shimadzu 6000 X-ray diffractometer in the reflection mode which had a graphite monochromator and a Cu K α radiation source (0.954Å) at a scan rate of 5.0000 (deg/min) and scan range 5.000 - 65.000^{o} . The conditions for carrying out the analysis were: Radiation source: Cu K α; Voltage: 40Kv; Current: 30mA; Scanning with step of 0.02°; Scanning, Time rate: 5°/min; Interval of scanning: 5 to 60° (2θ) consecutively. Start: 10.000 ° - End: 60°, Step: 0.02°, Step time: 0.3s, wavelength: 1.5406m and Temperature: 25^{o}.

3.5.8 Determination of water absorption behaviour of composites

In order to evaluate the absorption efficiency of the polymer composite of this research, 1g each of PP/$CaCO_3$, PP/CNSP and the neat polypropylene sample at the different filler loading were subjected to sorption test for 72hrs (i.e. three days).

The water absorption behavior characteristics of PP/$CaCO_3$ and PP/CNSP composite were studied and determined by immersing the composites of the different compositions in distilled water at room temperature for 72hrs. The test specimens (30mmx30mm) were cut and weigh measure accurately from the composite and tested for the water absorption capacity and strength

according to ASTM D570. The conditioned composites were taken out after 72hrs and then wipe with tidy tissue paper to free the sample from surface water. They were then weighed again to determine the weight gained. The percentage (%) sorption of water was calculated by the formula:

% sorption of water = $\frac{S1-SO}{SO} \times 100\%$

Where; S_1 = final weight of the composite

S_O = initial weight of the composite

CHAPTER FOUR

4.0 RESULTS

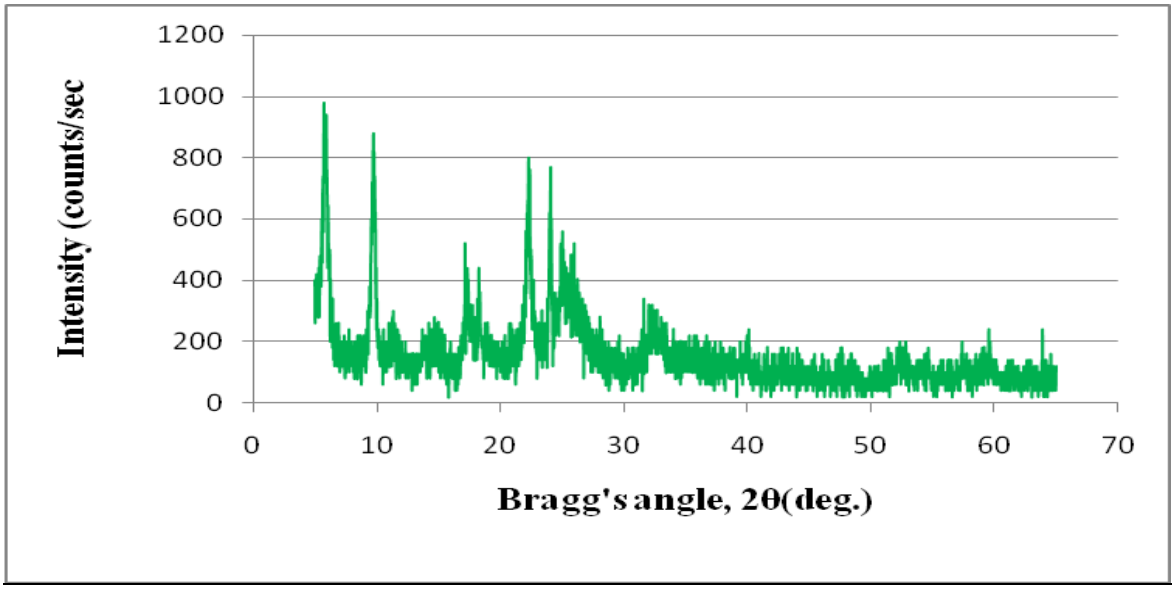


Figure 4.0: X-Ray Diffraction Pattern of Cashew Nutshell Powder

4.1 Result of Tensile Strength

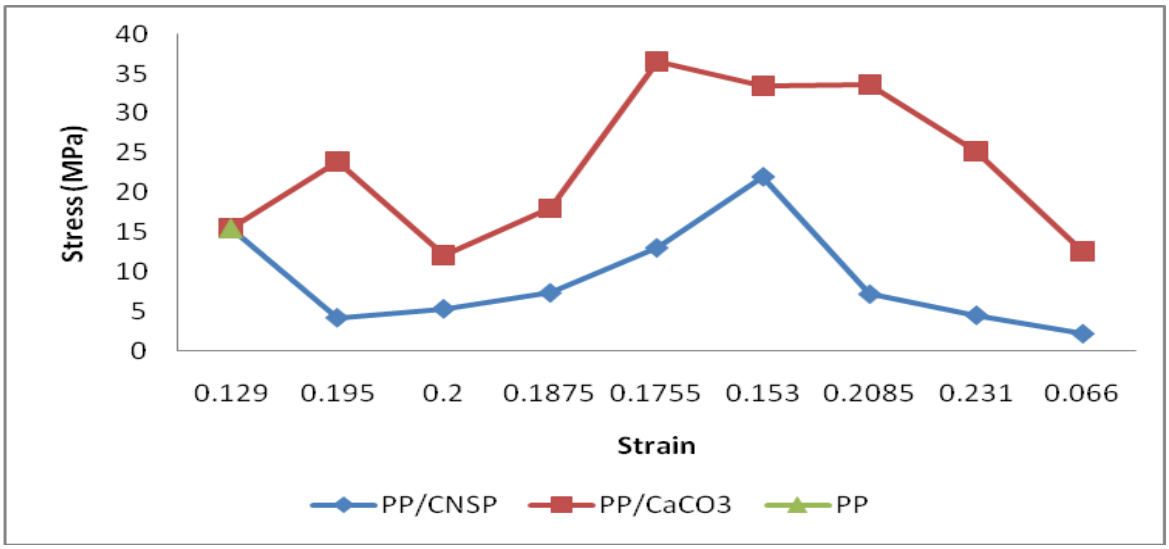


Figure 4.1: Stress-Strain Curve of PP, PP/$CaCO_3$ and PP/CNSP composites

4.2 Effect of Filler Loading on the Stress of the Composites Prepared

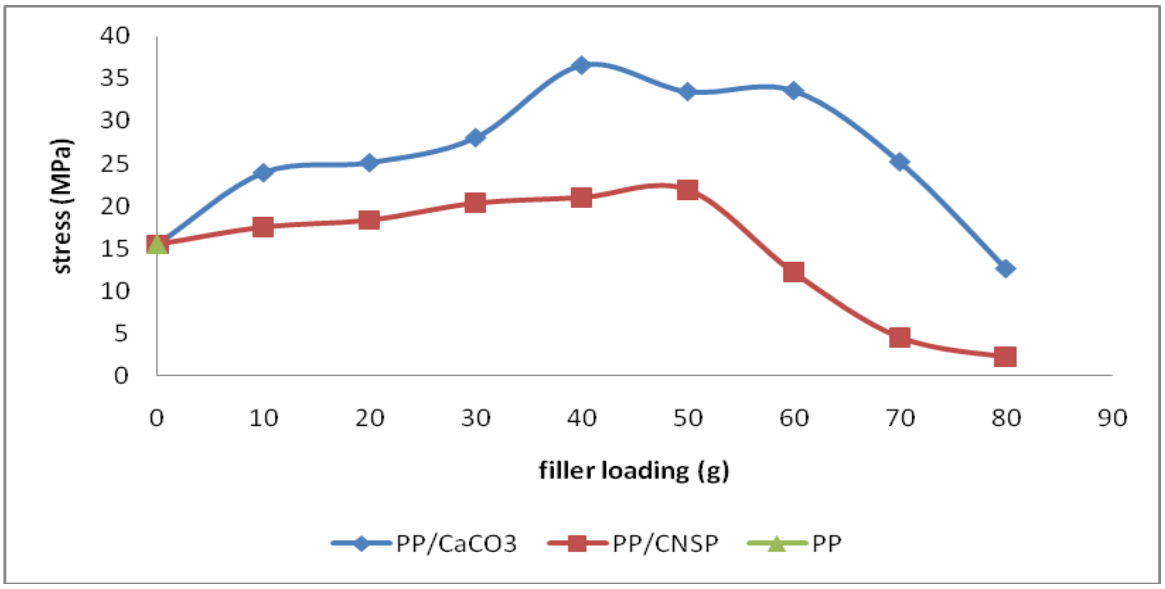


Figure 4.2: Effect of filler on the stress of PP, PP/$CaCO_3$ and PP/CNSP composite

4.2 Result of Elongation at break

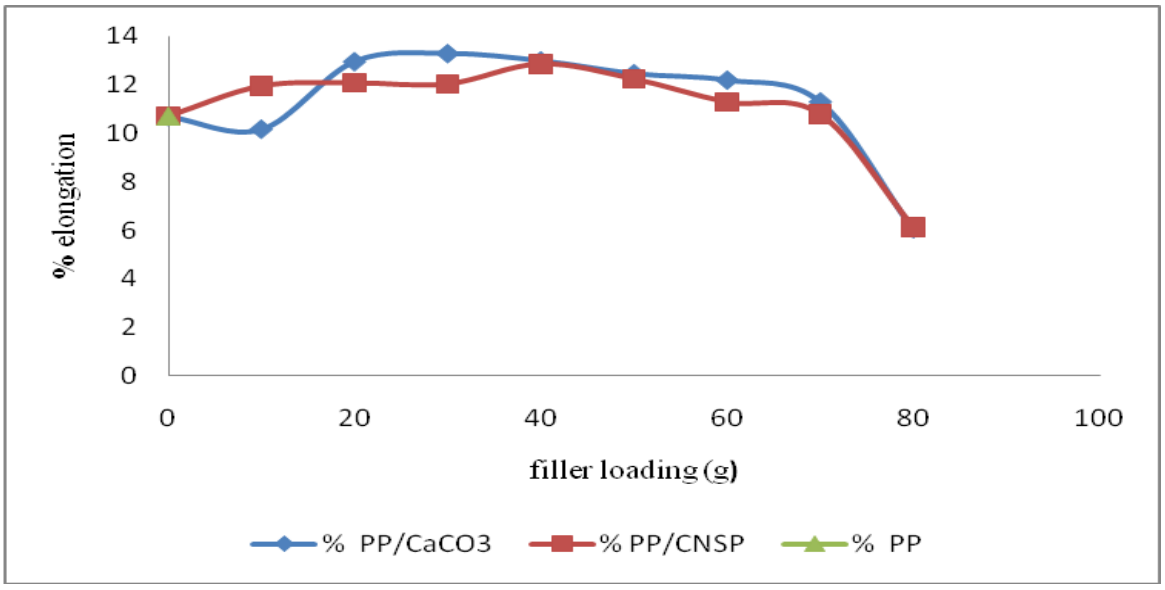


Figure 4.3: % elongation of PP, PP/$CaCO_3$ and PP/CNSP composites

4.4 Result of Young's Modulus

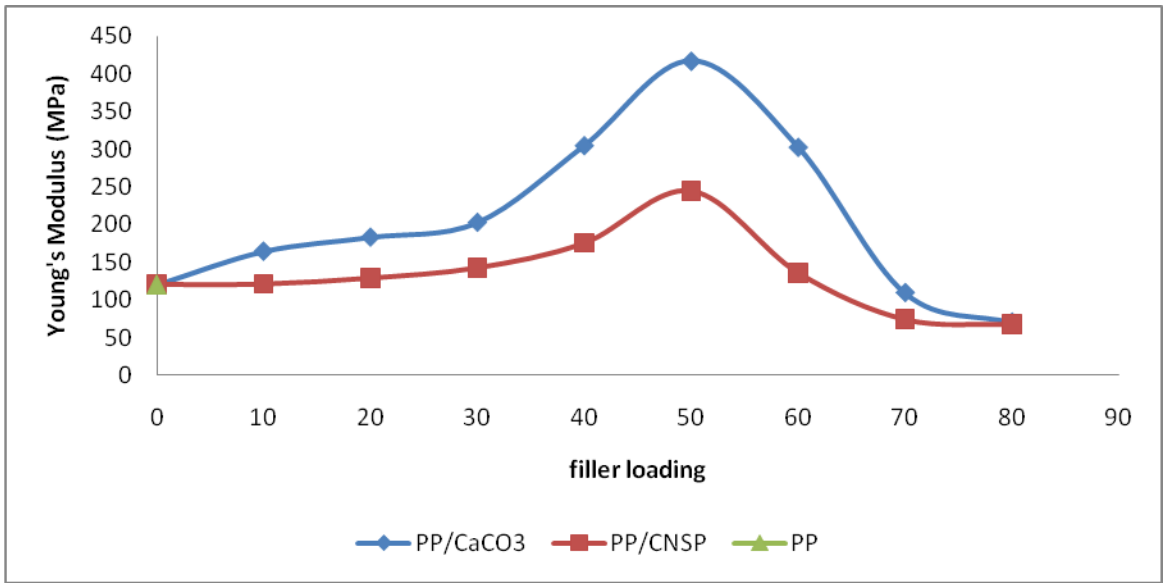


Figure 4.4: Young's Modulus of PP, PP/$CaCO_3$ and PP/CNSP composites

4.5 Impact strength results

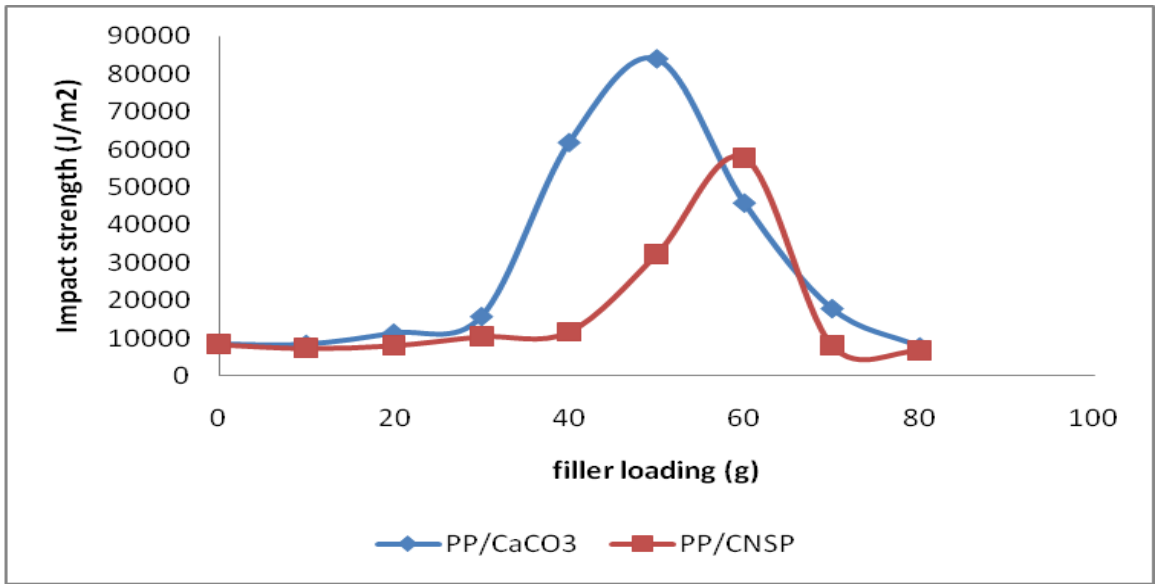


Figure 4.5: Effect of filler content on impact strength of PP, PP/$CaCO_3$ and PP/ CNSP

4.6 Hardness Results

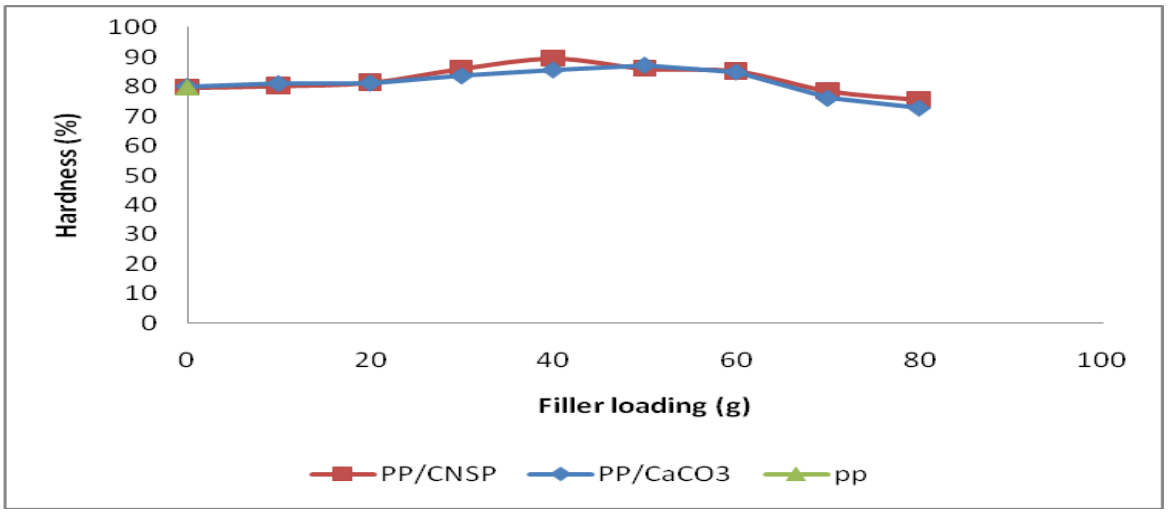


Figure 4.6: Effect of filler content on the Hardness of PP, PP/$CaCO_3$ and PP/CNSP composites

4.7 Sorption Result

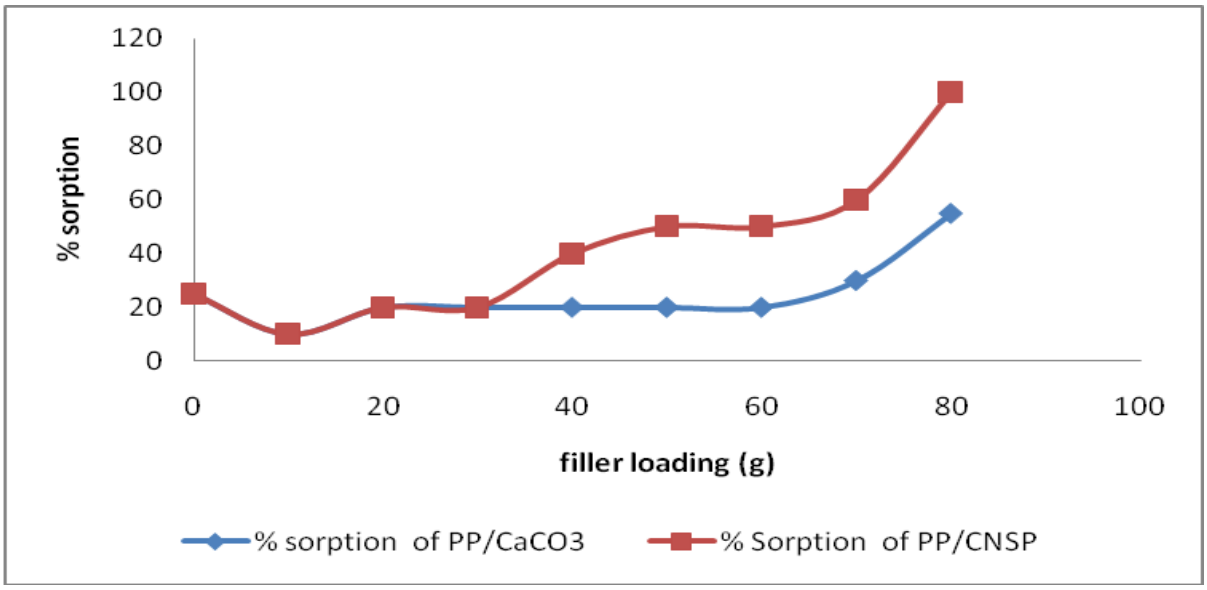


Figure 4.7: Effect of filler content on the sorption of PP, PP/$CaCO_3$ and PP/CNSP composites

4.7 Morphology Results

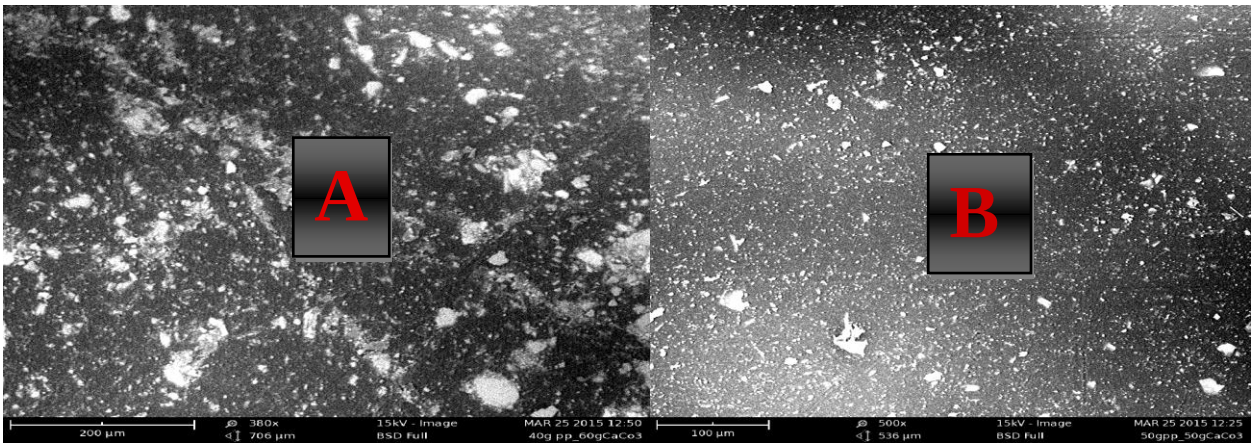


Plate I: SEM micrographs of composite (A) 60/40 PP/$CaCO_3$ and composite (B) 60/40 PP/CNSP

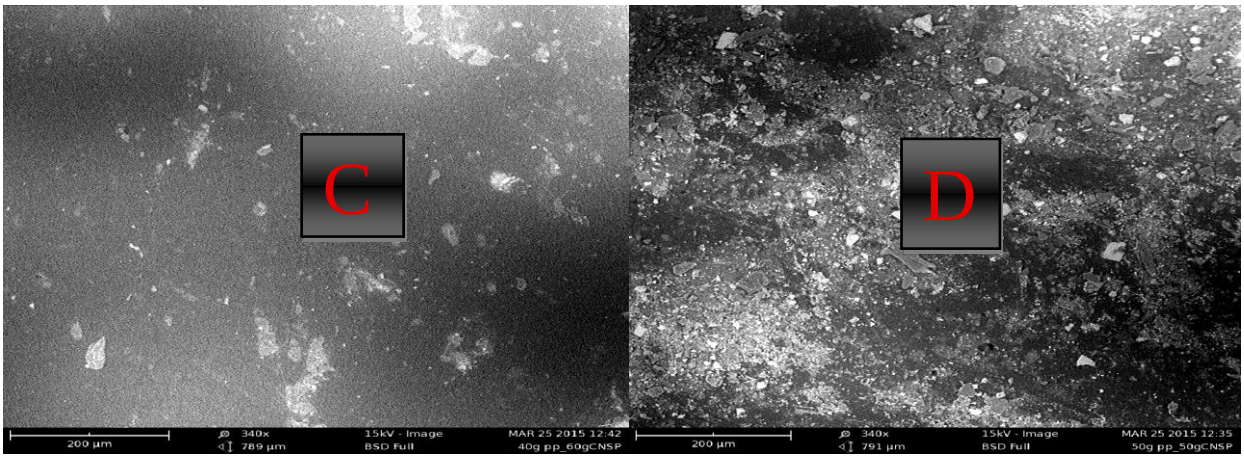


Plate II: SEM micrographs of composite (C) 50/50 PP/$CaCO_3$ and composite (D) 50/50 PP/CNSP

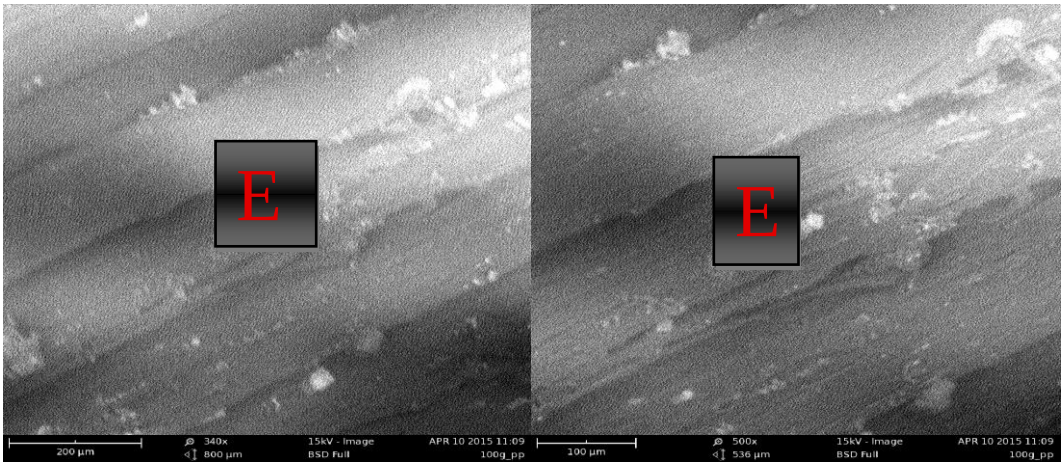


Plate III: SEM micrographs of (100PP/100PP) polypropylene

Figures 4.8a-4.8e represent the XRD pattern from Bragg's angle of 2θ 5^{o} – 55^{o} showing intensity against 2θ angle.

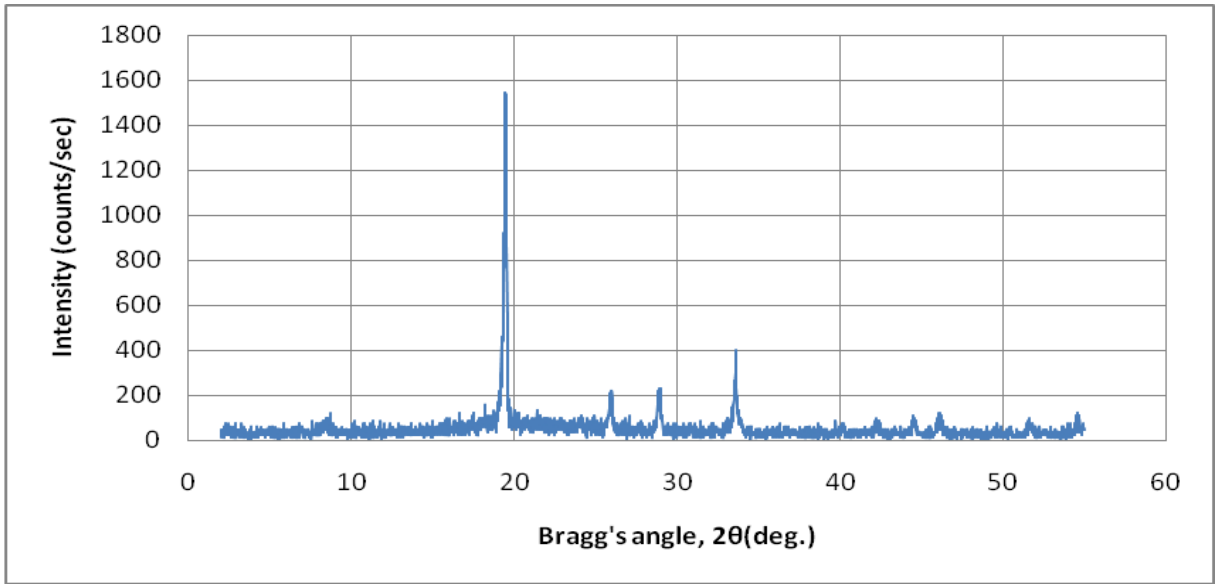


Figure 4.8a: XRD pattern unfilled polypropylene

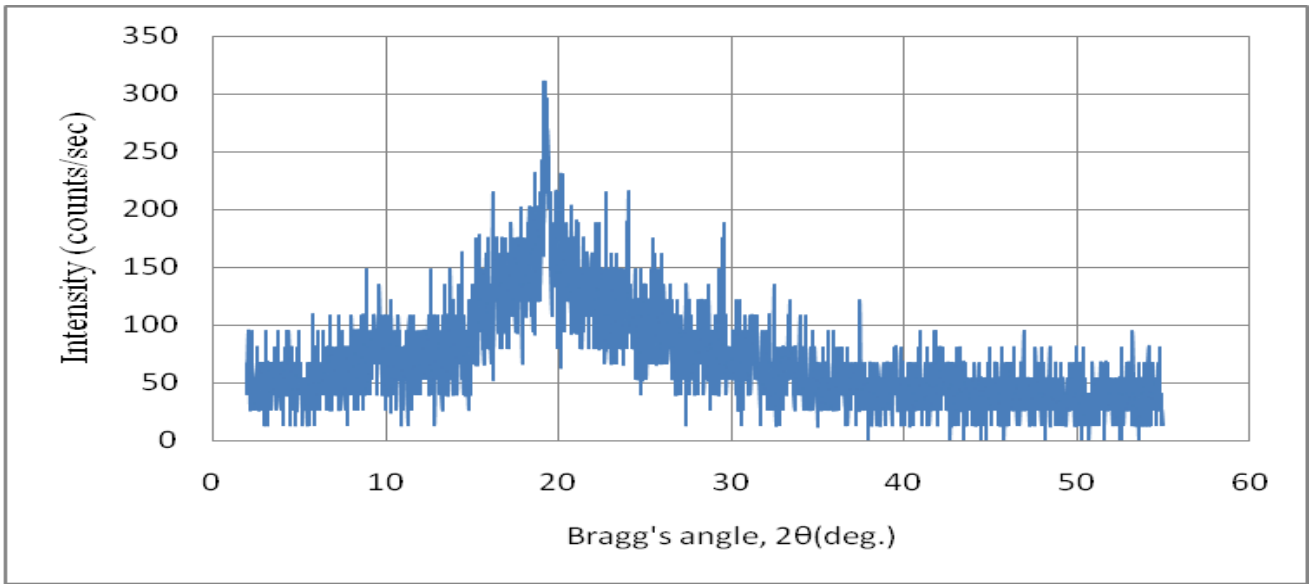


Figure 4.8b: XRD pattern of 60/40 PP/CNSP composites

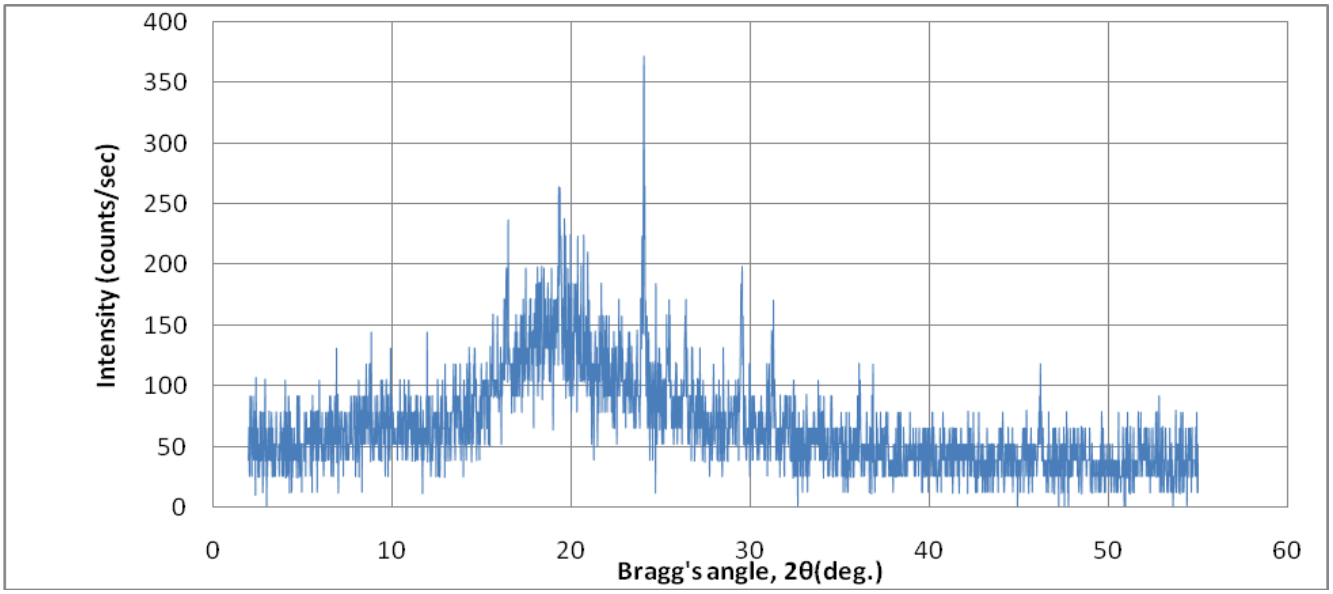


Figure 4.8c: XRD pattern of 50/50 PP/CNSP composites

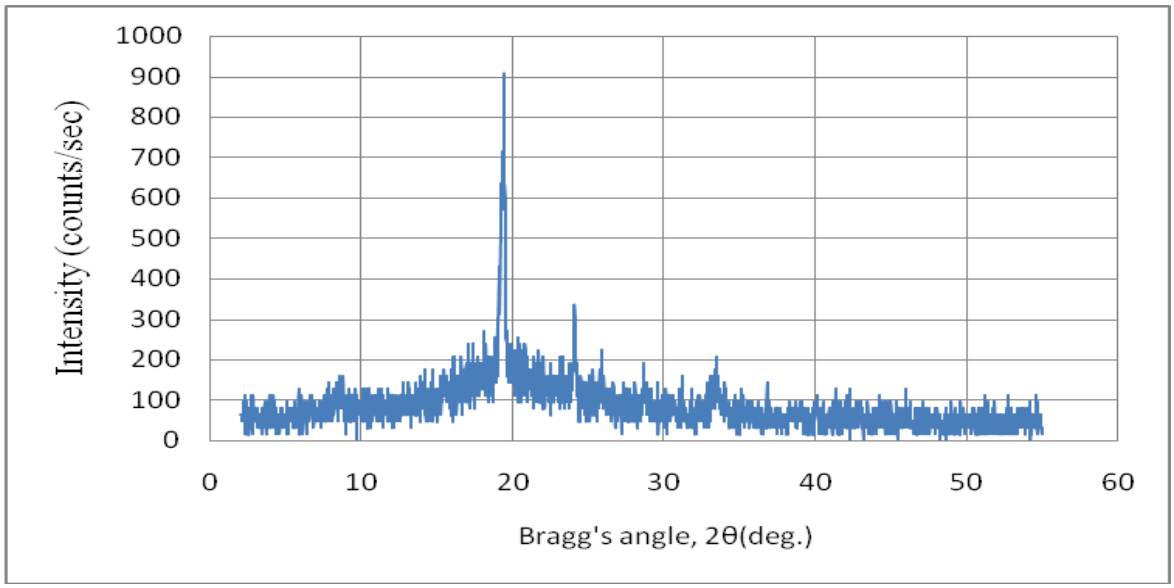


Figure 4.8d: XRD pattern of 60/40 PP/$CaCO_3$ composites

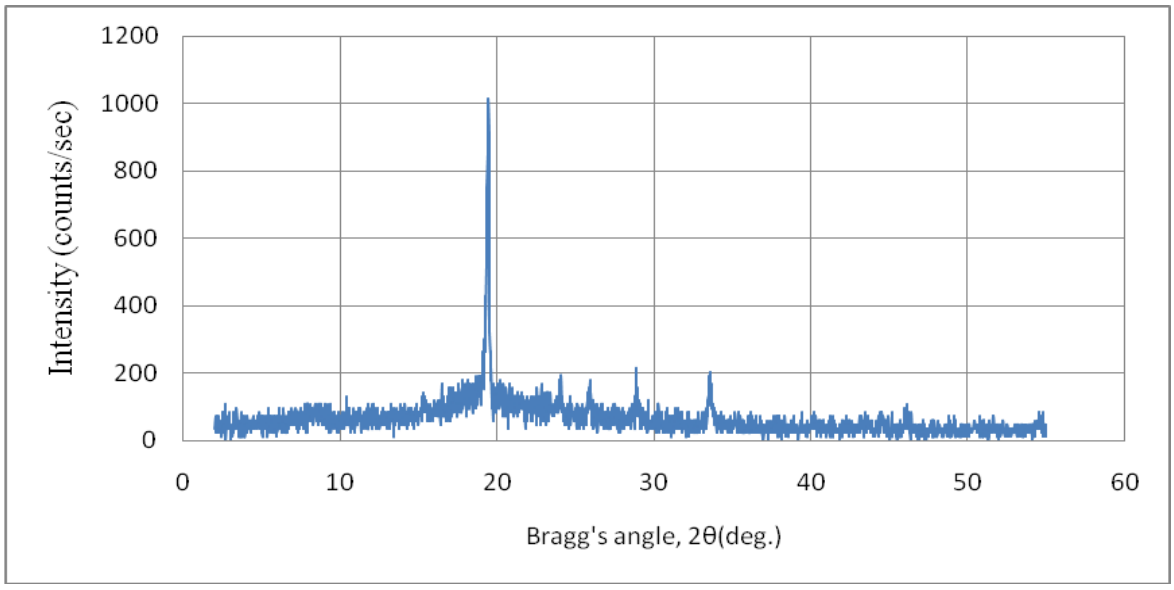


Figure 4.8e: XRD pattern of 50/50 PP/$CaCO_3$ composites

CHAPTER FIVE

5.0 DISCUSSION OF RESULTS

5.1 Structural Characterisation of Cashew Nutshell Powder

The powder X-ray diffraction spectrum of samples is shown in Figure 4.0. The diffractograms of the cashew nutshell powder samples exhibited diffraction patterns, with diffraction peaks of the 2θ angles at 10°, 20°, 30°, $40^{o,}$ 50^{o} and 60^{o} which can be assigned to the 30-1836, 29-1129, 33-0191, 34-0394, 35-0816 and 36-1249 reflections, respectively.

5.2 Tensile Strength

The results of tensile tests (stress-strain curve) for the PP/$CaCO_3$ and PP/CNSP are presented in Figure 4.1. The results show that the unfilled polypropylene has lower tensile strengths than both PP/$CaCO_3$ and PP/CNSP so, they have higher tensile strength and higher elongations as compared to pure polypropylene, and on the other hand these figures show the behaviour of both types of composites which are intermediate between the pure polypropylene. By increasing the weight of the two fillers, that is, the calcium carbonate and the cashew nut shell powder from 10 g up to 50 g to the PP, there was increase change from soft and brittle to hard and tough with increase in percentage of PP in the composite.

It was also noted that the samples of ratio 50/40 PP/$CaCO_3$ and 50/ 40 PP/ CNSP had the highest tensile stress meaning that, they can with stand maximum load at a particular applied load as compared to samples of other ratios. These specimens could bear loads of 1075 N and 468 N with extensions of 4.44 mm and 6.12 mm respectively. The stress-strain curve showed a sharp increase with increasing load before reaching the maximum stress and then irregularities and

staggered decrease in stress was recorded for both conventional ($CaCO_3$) and new composites (CNSP) as reported by (M.K. Hossain *et al*., 2011).

There was sharp decrease of the mechanical properties on further addition of fillers in to the polypropylene matrix with (80/20) of both the calcium carbonate and cashew nutshell powder having the least mechanical properties. Levita *et al.*, (1989) indicated that nano $CaCO_3$ treated with titanate coupling agent helped to raise the fracture toughness of polypropylene. The increase in modulus can be explained by the filler presence in PP that limits mobility and deformability of the matrix through the introduction of mechanical restraint in the composites system agreeing with observation by Guerrica-Echevarriâa, (1998).

Other authours studied the mechanical and morphology of PP/$CaCO_3$ nanocomposites and suggested similar results as reported in this work.

Calcium carbonate and Cashew nutshell powder reinforcement effect on the tensile strength and Young's modulus of polypropylene matrix is demonstrated. As depicted in the figure 4.1 and Figure 4.2, the tensile strength increased linearly up to 50 g weight, after that it tends to decrease despite the further rise in the weight of the fillers. This may be associated with the fact that, as the weight of the fillers increased. The weak interfacial area and the micro spaces increased between the filler and the matrix, thereby reducing the tensile strength. It is also observed, at high filler loading, it is more difficult for the resin to fully impregnate the filler thus leading to poor interfacial bonding and consequently lower mechanical properties. Thus, poor wetting results in poor stress transfer efficiency across the fillers' surfaces, which leads to agglomeration, and stress transfer gets hindered. As a result, there is a decreasing trend in tensile strength with increasing filler content in the composite. Moreover, as shown in figure 4.4, the Young's modulus increased with the increasing of weight of both $CaCO_3$ and CNSP, this may be due to

the further rise in the weight of filler in to the polypropylene matrix, which reduces the matrix mobility, which increases the stiffness of the composite. Sangthong *et al*., (2009) and El-Shekeil *et al.*,(2012).

Wang *et al.*, (2002) reported that the mechanical properties of PP, especially the ductility, were effectively improved by the incorporation of nano-$CaCO_3$ pretreated with stearic acid in an ultra-high-speed mixer (6000 rpm).

Ma *et al.*, (2005) showed that, for PP nanocomposites filled with grafted nano-$CaCO_3$ particles, a significant increment in notched impact strength and elongation at break of PP at a rather low content of nano-$CaCO_3$ was found. Meanwhile, the tensile stiffness of the composites was also slightly increased, and the yield strength of the composites was almost unchanged. It can also be noted from the figures that, both the Young's Modulus and the elongation properties decreased with increment of the two fillers loading with decrease in the polypropylene content due to the fact that polypropylene could not melt the dispersed filler at higher loading which made it less strong and more brittle compared to samples of lower ratios.

5.3 The impact strength

The Impact strength or toughness is often the deciding factor in material selection because, impact test measures the ability of a polymer to withstand load imposed upon it when struck by an object at high velocity. The impact resistance of a material is its ability to withstand shock. It is characterized by the impact strength, which is the point at which the material breaks or yields under an applied load. The impact strength of a polymer depends upon the inherent properties of such a polymer, condition of fabrication, design of the product, environmental conditions, and the nature and frequency of shock. The conditions of fabrication are more frequently responsible for failure than any other impact failure (Ohikhena, 2002).

Figure 4.5 Shows the effect of filler ratio on impact strength for PP/$CaCO_3$ and PP/CNSP respectively, there was increase in impact strength in both samples as compared with the neat pp, however, pp/ $CaCO_3$ had the highest impact strength values as compared to PP/CNSP and the pure pp. It was obvious from figure 4.5, that there was increase in the impact strength as $CaCO_3$ and CNSP were incorporated into the pp, as the filler content increased with decrease in the weight of PP, that is, up to 50/50 for both PP/$CaCO_3$ and PP/CNSP respectively; as can be seen from Figure 4.5. A depression in impact strength as the filler content increased when mixed with pp was noted, after the maximum impact strength was obtained and depression ratio decreased with increase in filler content. The optimum impact strength was obtained at 50 g filler loading for both calcium carbonate and cashew nutshell powder and the minimum impact strength at 80 g filler loading of the fillers.

Calcium carbonate ($CaCO_3$) is one of the most commonly used inorganic filler in PP so many groups have studied the mechanical properties of PP/$CaCO_3$ nanocomposites. Chan *et al.,* (2002) found that impact strength of PP could be increased from 55 J/m to 133 J/m by adding 9.2 vol. % surfactant treated 44 nm $CaCO_3$.

Chen *et al.,* (2004), studied toughening of propylene-ethylene copolymer with nano-$CaCO_3$, and found that the toughness of the matrix substantially improved. At a nano-$CaCO_3$ content of 12 phr, the impact strength of composite at room temperature reached 61.6 kj/m^2, which was 3.02 times that of unfilled matrix. Kun Yang, *et al.,* (2005) studied the morphology and mechanical properties of polypropylene/calcium carbonate nanocomposites, he revealed that the intrinsic toughness of PP matrix would influence the toughening effect of the nanoparticles. Only in the case of the moderate matrix toughness (PP matrix), the nanocomposites could receive the highest extent of toughness increase (4.5 times that of matrix).

5.4 Hardness

The results were tabulated in Table 3. Hardness of (PP/$CaCO_3$ and PP/CNSP), the hardness test results confirmed that pure PP has lower hardness value than both PP/$CaCO_3$ and PP/CNSP composites having optimum hardness value for 60/40 (PP/$CaCO_3$) and 50/50 (PP/CNSP). Furthermore, it was also noticed that the hardness values at (PP/$CaCO_3$) and (PP/CNSP) composites increased as the weight of both fillers increased from 10g to 50g for PP/CNSP and 10 g-60 g for PP/$CaCO_3$ loading and slightly decreased from 60g to 80g for PP/CNSP and 70 g-80 g for PP/$CaCO_3$ loading. This was related to the nature of the polypropylene, calcium carbonate and the cashew nut shell powder. The increased rate of the hardness for the PP/$CaCO_3$ was higher than PP/CNSP this is due to the difference between both types of PP/$CaCO_3$ and PP/CNSP molecular structure. From this curve, the highest value was recorded at 50/50 (PP/$CaCO_3$) composite which was $93^0/_0$.

5.5 Statistical Analysis of Impact Strength

In order to ascertain the Impact strength obtained for proposed filler (cashew nutshell powder) whether it could be substituted in place where the conventional commercially available filler (calcium carbonate) is applicable. The impact test results obtained were subjected to statistical Paired t- test, (Table 10) in the Appendix. The results were as follows:

$$t_{cal} = \frac{\bar{Q}}{Sd} x\sqrt{N}$$

$$S_D = \sqrt{\frac{\Sigma(Qi-\bar{Q})2}{N-1}}$$

where: t_{cal} = calculated t value, $\bar{Q}$ = mean average of impact strength values, Sd = standard deviation N = number of impact strength values recorded. The tabulated t value at 98% confidence level for eight degrees of freedom is 2.90. therefore, $t_{cal} = 2.680 < t_{table,}$ and therefore

there is no significant difference between the two fillers at this confidence level, hence, the null hypothesis is valid and the new filler (cashew nutshell powder) can be used in application where the existing filler (calcium carbonate) is used.

5.6 Effects of modification of unfilled and filled PP, PP/$CaCO_3$ and PP/CNSP Composites on equilibrium sorption

Tables 8-9 give the sorption data of the unfilled polypropylene (PP) and filled PP/$CaCO_3$ and PP/CNSP composites in water. The % absorption values were plotted against the various compositions in Figure 4.7 which gives the sorption graph of the sorption test of the polymer matrices. Sorption data in water (H_2O) gave a weight gain, the polymer composites (both unfilled and filled) witnessed a weight gain due to their absorption of water. However, solvent absorption increases with filler content. The explanation for this is that the water molecules slowly diffused into the polymer composites structure leading to high polymer-polymer and polymer – solvent intermolecular forces as a result of strong hydrogen bonding as observed by Aithal (1990). Three different mechanisms have been proposed for moisture penetration into the composite (two or more physically distinct and mechanically separable materials). The main process is the diffusion of water molecules inside the microgaps between the polymer chains. The other two mechanisms are capillary transport of water into the gaps and flaws created at the interface of fibre and polymer matrix because of incomplete wettability and impregnation and also diffusion of water molecules into the micro-cracks formed in the matrix during the compounding process (Lin, 2002). There was gradual increased of the percentage sorption of water from unfilled polymer matrix to polymer composites in which we observed that for composition of 90/10, 80/20, 70/30, 60/40, 50/50, 40/60 there was gradual rise of water absorption as the filler ratio increases for PP/CNSP while the sorption data for 30/70 and 20/80 PP/CNSP compositions showed large

increase of the percentage of absorption of water. This observation revealed that, interaction has taken place between the polypropylene matrices and the cashew nutshell powder filler which might have altered the molecular order of PP/CNSP composites at these compositions compared to those cases of the same compositions for PP/$CaCO_3$. Although, in the case of PP/$CaCO_3$ the constant values of sorption observed for 80/20, 70/30, 60/40, 50/50 and 40/60 revealed that no interaction has taken place to have altered the molecular order of PP/$CaCO_3$ composites composition compared to the case of PP/CNSP (Table 9 and 10) whereas increase in the weight of the $CaCO_3$ in polypropylene matrix increases the % sorption of water indicating slight change of molecular order at higher filler loading (both $CaCO_3$ and CNSP) even though the increase was not as much as for PP/CNSP compositions.

Similar result has also been reported by Mamza, (2011) when sorption test was conducted using four different solvents (i.e dil. HCl, NaOH, CH_3COCH_3 and H_2O) on PS/PVAc blends (both unfilled and filled), it was reported that in all the four solvents, sorption increased, except acetone showed a dramatic decline in % absorption as the filler loading was increased. The equilibrium of absorbed solvents can be attributed to the level of polymer-polymer interactions. In the study, acetone seemed to dissolve the crystalline portion of the blends and could not be used to detect the differences between the polymer blends and their molecular structure, while water, HCl and NaOH which can swell the amorphous regions of the polymer but will not dissolve the blend crystallites, can be used to detect differences between the polymer blends and their molecular structure (Mamza, 2011).

The subsequent rise in sorption values was attributable to the saturation of the gap between polypropylene matrix and the filler which assisted inter-grain friction in the melt, thereby increasing the gelation level. The amount of water absorption of the composites increased with

the incorporation of filler in the polymer matrix. The extent of solubilizing the composites is dependent on the nature of solvents used and the filler addition into the polymer composites structure (Figure 4.7). It is evident from these results that the polymer composites – solvent interactions were more dominant in the presence of the solvents over polymer –filler interaction. It was also therefore established further for this study that sorption parameters of PP/$CaCO_3$ and PP/CNSP composites were largely dependent on filler types and the dependence were in the order, calcium carbonate> cashew nutshell powder. The PP/CNSP had the highest % water absorption for the filled polypropylene composites but a general trend of increase in % water absorption was observed for all polypropylene-filled composites for both or all fillers (i.e. $CaCO_3$ and CNSP) except for PP/$CaCO_3$ that gave a constant % of absorption of water due to the reasons earlier deduced.

It can be established here that Sorption can be used to detect the structural changes in the PP/$CaCO_3$ and PP/CNSP composites this is because of the higher adhesive force between PP and $CaCO_3$ as compared to that of PP and CNSP

5.6.4 Morphological studies on the PP, PP/$CaCO_3$ and PP/CNSP composites

The purpose of this analysis was to investigate the dispersion of calcium carbonate or cashew nutshell powder in polypropylene. Excellent dispersion of CNSP was observed (Plate I). Agglomerations in the polypropylene matrix were observed from the micrographs (Plates I-III) of infused polypropylene samples prepared through mechanical mixing and thermal pressing methods, respectively.

These agglomerate areas create stress concentration zones which might act as hardness initiators. Strong attractive van der Waals forces cause calcium carbonate to agglomerate, which reduces the strength of the nanocomposite by stress concentration effect. Agglomerates are called

nanoropes, and are difficult to separate and infiltrate with matrix. They entangle and form nestlike structures due to their curvature and high filler ratios. Both disagglomeration and dispersion in resins depend on the relative van der Waals forces, curvature, and on the relative surface energy of the two fillers (either CNSP or $CaCO_3$) versus that of the resin.

High magnification (5000x) SEM micrograph in Plate I clearly exhibited that both fillers at these weight ratios were uniformly embedded in the 60 g polypropylene resin system. It could also be easily observed that the interfacial bonding between the CNSP or $CaCO_3$ and matrix was very compact which would allow the filler to be anchored in the embedding matrix. In essence, these fillers were likely to interlock and entangle with the polymer chains in the matrix as reported by Li *et al.,* (2008).

Mamza *et al.,* (2009) revealed that the morphological arrangements of PS/PVAc blends were studied by scanning electron microscopy and concluded that some compositions appeared to have surface inversion and that the structure-property relationships are essential aspects of polymer blend technology through proper end–use applications in the industries.

Plates I-III show two phase system, in order to evaluate the changes in surface morphology of the pure polypropylene to the polypropylene filled with the calcium carbonate and the cashew nutshell powder, pure polypropylene moulded sample in which no filler was incorporated, 100/0 PP, 50/50 PP/$CaCO_3$, 50/50 PP/CNSP, 40/60 PP/$CaCO_3$ and 40/60 PP/CNSP were subjected to scanning electron microscopic analysis (SEM) in order to know the actual interaction between the polypropylene and $CaCO_3$, polypropylene and the cashew nutshell powder.

The SEM micrographs clearly show that the difference of the morphology of the polypropylene filled composites (50/50 PP/$CaCO_3$), (60/40 PP/$CaCO_3$), (50/50 PP/CNSP) and (60/40

PP/CNSP) composites are compatible and there was high degree of miscibility. That is, the adhesion between the fillers and the polypropylene was clearly observed.

However, the compatibility decreased with filler loading (both $CaCO_3$ and CNSP) these owned to the less interaction between the pp and both fillers of those compositions as a result of the disorderliness of the structural molecular arrangement of the repeating units in the polypropylene chain that occur at higher filler dosage.

Thus, addition of CNSP or $CaCO_3$ in to the polypropylene matrix enhanced the polymer chains by providing better interfacial bonding between the polypropylene and the fillers (CNSP or $CaCO_3$) and some mechanical properties such as tensile, elongation at break, impact strength, and hardness of the polypropylene were improved which may be applicable for certain industrial end-use applications

5.6.5 Structural Characterisation of the Polypropylene Composites prepared

XRD analysis was employed to ascertain the effect of filler on the crystallinity of the polypropylene composites. The results of the comparative XRD analysis of the polypropylene composites and the neat polypropylene were illustrated in Figures.4.8a-4.8e and are summarized in Table 11. The following changes in XRD patterns were observed (a) increase in d spacings of the Polypropylene composites of fillers content 40 g and 50 g (both cashew nutshell powder and calcium carbonate) compared with the polypropylene without filler, for instance the polypropylene without filler had d-spacing value of 2.66410Å at $2\theta=33.6130^{o}$ compared with 2.84384Å at $2\theta=31.4316^{o}$ and 2.67443 at $2\theta = 33.4794^{o}$ for both 60/40 PP/$CaCO_3$ and 60/40 PP/CNSP respectively, (b) shift of peak positions to lower 2θ values can be seen in the polypropylene composites dosed with fillers compared with the unfilled or the unmodified polypropylene and (c) Physical changes can also be observed in the XRD parameters as the two

fillers loading increased in the polypropylene composites. The crystallinity index was calculated and the highest value was obtained as (94.82%) for the unfilled PP compared to the filled composite (Table 11). In the present work, the crystallite size was calculated from the observed XRD pattern of different compositions of 60/40 PP/$CaCO_3$, 60/40 PP/CNSP and the pure Polypropylene, respectively. The crystallite size of the composites can be calculated from the full-width at half-maximum (FWHM) measurement for the prominent X-ray diffraction peaks using Scherrer formula,(equation 1) (Srivastava, 2011).

From the observed results, the XRD patterns (prominent peak) of samples PP/$CaCO_3$ and PP/CNSP showed that, both composites materials have more of amorphous regions than crystalline regions, that is, the incorporation of the two fillers in to the neat polypropylene decreased the crystallinity of the polypropylene with increase in filler loading.

In Figure 4.8a-4.8e, XRD patterns for unfilled PP, filled PP/$CaCO_3$ and PP/CNSP (Table 11) show strong diffraction peaks at 19.5208^o, 33.6130^o,19.2451^o, 19.3648^o, 29.5839^o, 31.4316^o, 24.0683^o, 29.5125^o, 23.9400^o, 19.3631^o, 24.1144^o, 33.4794^o and 19.3889^o, 33.5275^o, 28.8550^o respectively. The XRD patterns show sharp and well defined peaks (the prominent peaks), indicating the crystallinity of the composites produced (Figure 4.8a - 4.8e). Intensity of diffraction peaks for the composites PP/CNSP was lower than both of the pure PP and PP/$CaCO_3$ of composites. This implies that the intensity depends on the composition of the composites, indicating certain interaction between the polypropylene and the fillers incorporated. Similar result was reported by Sumit *et al.*, (2013), when he carried out study of XRD pattern of mixed composite of $MgTiO_3$ and ZnO prepared by the of synthesis of magnesium titanate ($MgTiO_3$) composite produced by Mg and Ti material with ZnO.

CHAPTER SIX

SUMMARY, CONCLUSION AND RECOMMENDATIONS

6.1 Summary of Results

Results of the various tests performed were analysed, values obtained were evaluated and averages were calculated, mechanical properties were improved for the polypropylene composites compared to the neat polypropylene as the two fillers were incorporated in to the polypropylene matrix with optimum mechanical properties such as tensile strength, % elongation at break, hardness behaviour and impact strength at 50/50 for both PP/$CaCO_3$ and PP/CNSP composites compared to samples of other ratios.

It was also noted, that the polypropylene composites samples have good % sorption except 20/80 PP/CNSP which had a poor % sorption. The X-ray diffraction analysis showed that, the incorporation of the two fillers in to the neat polypropylene decreased the crystallinity of the polypropylene and the crystallinity decreases with increasing filler loading.

6.3 Conclusion

The present study has generally investigated the effect of the filler content in the polypropylene on the mechanical properties of PP, PP/$CaCO_3$ and PP/CNSP and the results were as following:

1. The mechanical properties of PP/$CaCO_3$ gave the highest values compared to PP/CNSP and PP composites.
2. the mechanical properties such as (tensile strength, % elongation, young modulus, impact resistance, hardness) of both PP/$CaCO_3$ and PP/CNSP composites increased with increment of filler weight content (10-50 g) and decreased on continuous addition of both of the fillers, with drastic reduction of the mechanical properties at (70 g and 80 g) fillers weight except hardness that slightly decreased at all the filler loading (10-80 g). It has been noted that (50/50) of PP/$CaCO_3$ and PP/CNSP appeared to withstand high loads as they were compared to other samples of other ratios.
3. Both 60/40 PP/$CaCO_3$ PP/CNSP and 50/50 PP$CaCO_3$, PP/CNSP are completely compatible at which there are no phases that are grossly separated.
4. The impact resistance of the cashew nutshell powder was ascertained by subjecting the impact test results to statistical analysis.
5. It has also been noted that the %Sorption of the composites samples were comparable at (10-50 g) filler loads, with (60-70 g) filler loads of 50% and (80 g) filler loads having 100% water absorption in case of cashew nutshell powder filler.
6. Null hypothesis was also verified with t_{cal} =2.680 and tabulated t value at 98% confidence level for eight degrees of freedom = 2.90

The results of the study also showed that both the addition of $CaCO_3$ filler and the cashew nutshell powder filler have resulted in the linear improvement in the mechanical properties of the

Polypropylene. The tensile strength, Young's modulus and elongation at break of PP were all increased with the incorporation of both the Calcium Carbonate filler and Cashew Nutshell Powder at different content of 10 g, 20 g, 30 g, 40 g and 50 g further increase of the two fillers loading in the polypropylene slightly decreased the above listed mechanical properties and sharp declined of the properties at 70 g and 80 g of fillers content.

6.3 Recommendations

Further work is recommended in the following areas:

1. There is need to conduct research on these new fillers at different particle sizes in order to evaluate their abrasion resistance
2. Heat stabilizers should be impregnated in to the polypropylene processing samples to avoid depression of its mechanical properties at elevated temperature.

   Analysis should be carried out on the polypropylene composite at different temperatures in order to analyse their creep property
3. Flexural analysis should also be carried out on the polypropylene composites
4. Research should be performed with polypropylene blends as the polymer matrix and polypropylene composite to compare results.
5. Thermal characterisation using differential scanning calorimetry (DSC) of the different samples may aid in analyzing the interaction between the filler and the polymer matrix at high temperature.
6. Further research should also be performed on the polypropylene and polypropylene composites with the incorporation of biologically active agent in order to compare their susceptibility to microbial attack.

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APPENDIX

Table 1: Tensile Test results for the PP/CNSP Samples (Average Results)

S/No	Sample (g)	Average thickness (mm)	Average width (mm)	Area (mm^2)	Average force (N)	Stress (N/mm^2)	Average extension (mm)	Gauge length (mm)	Strain	Young Modulus (N/mm^2)
1	100pp	3.25	8.1	26.3	408	15.4986	5.16	40	0.129	120.14399
2	100pp			25						
3	10CN	2.65	8.05	21.3	468	21.9384	6.12	40	0.129	143.38795
4	10CN			325						
5	20CN		7.95	29.8	126	4.22642	7.8	40	0.195	21.67392
6	20CN	3.75		125						
7	30CN	4.6	8.05	37.0	198	5.3470	8	40	0.2	26.73507
8	30CN			3						
9	40CN	3.25	7.95	25.8	336	13.0044	7.02	40	0.1755	74.09888
10	40CN			375						
11	50CN	4.6	8.25	37.9	279	7.35178	7.5	40	0.1875	39.20948
12	50CN			5						
13	60CN	6.55	7.9	51.7	372	7.18910	8.34	40	0.2085	34.48009
14	60CN			45						
15	70CN	3.1	8.1	25.1	114	4.54002	9.24	40	0.231	19.65378
16	70CN			1						
17	80CN	2.75	9.85	27.0	60	2.21504	2.64	40	0.066	33.56127
				875						

Table 2: Tensile Test Results for the PP/$CaCO_3$ Samples (Average Results)

S/No	Sample (g)	Average thickness (mm)	Average width (mm)	Area (mm^2)	Average force (N)	Stress (N/mm^2)	Average extension (mm)	Gauge length (mm)	Strain	Young Modulus (N/mm^2)
1	100pp	3.25	8.1	26.325	408	15.4986	5.16	40	0.129	120.1431
2	100pp									
3	10Ca	4.2	8	33.6	804	23.9286	5.82	40	0.146	164.4575
4	10Ca									
5	20Ca	4.85		37.587						183.2070
6	20Ca		7.75	5	455	12.1051	6.9	40	0.173	
7	30Ca	4.5	8	36	650	18.0556	6.6	40	0.165	202.9079
8	30Ca									
9	40Ca	3.55	7.7	27.335	1000	36.5831	4.8	40	0.12	304.8595
10	40Ca									
11	50Ca	2.95	8.1	23.895	800	33.4798	6.6	40	0.165	417.1899
12	50Ca									
13	60Ca	4	8	32	1075	33.5938	4.44	40	0.111	302.6464
14	60Ca									
15	70Ca	4.1	8.2	33.62	425	12.64128	2.76	40	0.069	109.4276
16	70Ca									
17	80Ca	3.55	8.1	28.755	725	25.2130	1.95	40	0.049	70.17442
18	80Ca									

Table 3: Hardness Test Results

S/No	Filler loading (g)	PP/ $CaCO_3$ (%)	PP/CNSP (%)
1	0	79.71429	79.71429
2	10	81.14286	80.28571
3	20	81.28571	81.42857
4	30	83.71429	86.14286
5	40	85.71429	89.57143
6	50	87.28571	86.14286
7	60	85	85.28571
8	70	76.14286	78.57143
9	80	72.85714	75.57143

Table 4: Stress-Strain Table

S/No	Filler Loading (g)	PP/$CaCO_3$ (N/mm^2)	Strain	PP/CNSP (N/mm^2)	Strain
1	0	15.4985755	0.129	15.4985755	0.129
2	10	23.92857143	0.1455	17.53234511	0.195
3	20	25.10508813	0.1725	18.34701593	0.2
4	30	28.05555556	0.165	20.35177866	0.1875
5	40	36.58313517	0.12	21.00435414	0.1755
6	50	33.47980749	0.112	21.93835697	0.153
7	60	33.59375	0.111	12.1891004	0.2085
8	70	25.21300643	0.069	4.540023895	0.231
9	80	12.64128495	0.04875	2.215043839	0.066

Table 5: Young modulus of PP/$CaCO_3$, PP/CNSP and PP

S/No	Filler Loading (g)	Young modulus PP/$CaCO_3$ (N/mm^2)	Young Modulus PP/CNSP (N/mm^2)
1	0	120.1439961	120.1439961
2	10	164.4575356	120.653783
3	20	183.2070283	128.673924
4	30	202.9079242	142.209486
5	40	304.8594598	175.098884
6	50	417.1898756	244.387954
7	60	302.6463964	135.480098
8	70	109.4276094	73.5612703
9	80	70.17442393	66.7350967

Table 6: Percentage Elongation of PP/$CaCO_3$, PP/CNSP and PP

Filler loading(g)	S/No	% elongation (PP/$CaCO_3$)	%elongation (PP/CNSP)
0	1	10.69873523	10.69873523
10	2	10.16949153	11.94277421
20	3	12.96111665	12.0671781
30	4	13.29345297	12.02571014
40	5	12.99435028	12.85506946
50	6	12.46261216	12.23304997
60	7	12.1967431	11.27928675
70	8	11.29943503	10.78167116
80	9	6.048521103	6.116524984

Table 7: Impact Resistance of PP/$CaCO_3$, PP/CNSP and PP

S/No	Filler Loading (g)	Impact strength PP/CaCO3 (J)	Impact strength PP/CNSP (J)
1	0	0.25	0.25
2	10	0.25	0.22
3	20	0.35	0.25
4	30	0.45	0.3
5	40	1.8	0.96
6	50	2.5	1.64
7	60	1.3	0.36
8	70	0.5	0.23
9	80	0.23	0.2

Table 8: Sorption Test Results (PP: $CaCO_3$)

S/No	Filler loading(g)	Initial mass of PP: $CaCO_3$, S_o (g)	Final mass of PP: $CaCO_3$, S_1 (g)	Mass of water absorbed (g)	% Sorption Efficiency $\frac{S1-So}{So}$ x 100
1	0	1	1.25	0.25	25
2	10	1	1.2	0.1	10
3	20	1	1.2	0.2	20
4	30	1	1.2	0.2	20
5	40	1	1.2	0.2	20
6	50	1	1.2	0.2	20
7	60	1	1.3	0.2	20
8	70	1	1.55	0.3	30
9	80	1	1.25	0.55	55

Table 9: Sorption Test Results (PP/CNSP)

S/No	Filler loading(g)	Initial mass of PP/ CNSP, S_o (g)	Final mass of PP/CNSP, S_1 (g)	Mass of water absorbed (g)	% Sorption Efficiency $\frac{S1-So}{So}$ x 100
1	0	1	1.25	0.25	25
2	10	1	1.1	0.1	10
3	20	1	1.2	0.2	20
4	30	1	1.2	0.2	20
5	40	1	1.4	0.4	40
6	50	1	1.5	0.5	50
7	60	1	1.5	0.5	50
8	70	1	1.6	0.6	60
9	80	1	2.0	1.0	100

Table 10: Statistical Analysis of impact strength of PP/$CaCO_3$ and PP/CNSP

S/No	Filler Loading (g)	Existing filler ($CaCO_3$)	New filler (CNSP)	Q_i	$Q_i-\bar{Q}$	$(Q_i-\bar{Q})^2$
1	0	0.25	0.25	0	-0.3578	0.1280
2	10	0.25	0.22	0.03	-0.3278	0.1075
3	20	0.35	0.25	0.10	-0.2278	0.0665
4	30	0.45	0.30	0.15	-0.2078	0.0432
5	40	1.80	0.96	0.84	0.4822	0.2325
6	50	2.50	1.64	0.86	0.5022	0.2522
7	60	1.30	0.36	0.94	0.5822	0.3381
8	70	0.50	0.20	0.27	-0.0878	0.0077
9	80	0.23	0.20	0.03	-0.3278	0.1075
Total				3.22		1.2832
mean				$\bar{Q}$=0.3578		

Table 11: X-ray diffraction Analysis result

Sample	Peak number	Bragg's Angle, 2θ (deg.)	Bragg's spacing, d (Å)	I_{max} (count)	% of crystallinity (X_c)	Crystallinity Index, I_x (%)	Crystallites particle size, D (μm)
Unfilled	1	19.5208	4.54378	897.26			
polypropylene	2	33.6130	2.66410	1545.00	95.10	94.82	12.68
(100/0)	3	19.2451	4.60825	884.58			
Filled	1	19.3648	4.58003	192.53			
polypropylene	2	29.5839	3.01712	290.84	79.17	73.70	7.96
(60/40)	3	31.4316	2.84384	309.00			
Cashew Nutshell Powder							
Filled	1	24.0683	3.69458	128.85			
polypropylene	2	29.5125	3.02425	158.00	77.78	71.43	13.76
(50/50)	3	23.9400	3.71409	128.17			
Cashew Nutshell Powder							
Filled	1	19.3631	4.58043	523.41			
polypropylene	2	24.1144	3.68762	651.85	88.88	87.50	6.82
(60/40)	3	33'4794	2.67443	905.00			
Calcium Carbonate							
Filled	1	19.3889	4.57439	585.39			
polypropylene	2	33.5275	2.67070	1014.00	80.22	75.35	8.80
(50/50)	3	28.8550	3.09165	872.70			
Calcium Carbonate							

DETERMINATION OF T CALCULATED

$S_D = \sqrt{\frac{1.28323}{9-1}}$

$= \sqrt{\frac{1.28323}{8}}$

$= \sqrt{0.16040375}$

$= 0.4005$

Therefore, the $t_{cal} = \frac{0.3578}{0.4005} x\sqrt{9}$

$= \frac{0.3578}{0.4005} \times 3$

t_{cal},=2.680