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Research article

Evaluation of Electrical and Thermal Conductivity of Polymeric Wastes Doped with Activated Charcoal Produced from Doum Palm (*Hyphane thebaica* L.) Bark

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ABSTRACT

The growing concern over environmental sustainability and the need for innovative waste management solutions have spurred interest in the utilization of waste resources. This study investigates the potential of utilizing activated charcoal produced from Doum Palm (*Hyphane thebaica L.*) bark as a dopant to improve the electrical and thermal conductivity of polymeric waste materials. The doped materials were formed by incorporating varying proportions of activated charcoal into the polymer matrices through a melt-blending technique. The resulting samples were then subjected to certain characterization processes, including FTIR, SEM, Physico-Chemical analysis, and electrical and thermal conductivity testing. Physico-Chemical analysis is done to determine the activation efficiency of the activated charcoal produced. Electrical conductivity is evaluated to assess the potential of these doped materials for electronic applications and antistatic properties. Thermal conductivity measurements provide insights into their suitability for heat transfer applications. Polyethylene shows the highest electrical conductivity of 1.82×10^{-3} S/cm at 10wt% dopant concentration, with polypropylene performed best for thermal conductivity. The findings of this study contribute to the development of sustainable materials with improved properties from waste polymers. Hence, it is evident that on further modification they can be used for various applications, such as the development of conductive materials, used in electronics industries, contributing to the promotion of economy practices and reducing environmental impacts associated with polymer waste disposal.

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1. INTRODUCTION

Wastes are in various forms and types, and the existence, management and challenges in human settlements have been of concern to individuals, communities, governments, organizations, research and development (Rochman, 2013). In Africa, Nigeria, and the Northern region, the issue of plastic waste is particularly severe, that current statistics reveal the extent of the problem and highlight the urgent need for more effective waste management strategies to address this significant environmental challenge (UNEP, 2020). The best permanent way to address the menace of these wastes is by recycling. Doping can be employed to enhance their conductivity, so to be used for other purposes especially in industries for example producing capacitors, electrical appliances, diodes, plastic seats, cabinets etc. other than being disposed in the environment (Gupta et al., 2016).

Until about 48 years ago, in 1975 the notion that plastics could be made to conduct electricity would have been considered to be absurd. Polymers insulate and do not conduct electricity and so electric wires are coated with polymers to protect them and us from short circuits but Shirakawa et al., (1977) reinforced the then-emerging concept of organic materials behaving not as traditional insulators but as metals or semiconductors. This was particularly exciting because it created a new field of research on the boundary between chemistry and condensed matter physics, and because it created a number of opportunities. Because of that, conducting polymers (CPs) have gained increasing attention owing to their strong potential as alternatives to their inorganic counterparts, leading to significant fundamental and practical research efforts. In the late 1970s, many scientists considered CPs (or 'synthetic metals') to be intractable and insoluble. Since the discovery of polyacetylene in 1977 by Hideki Shirakawa, various important CPs have been investigated continuously, including polypyrrole (PPy), polyaniline (PANI), polythiophene (PT), poly(3,4-ethylenedioxythiophene) (PEDOT), trans-polyacetylene, and poly (p-phenylene vinylene) (PPV) (Hall, 2003). The structures of some conducting polymers are shown in Figure 1.

The conductivity of un-doped polymers can be increased by 10 or more orders of magnitude through doping. For instance, Janssen and Kemerink, (2013), reported the doping of polyacetylene with iodine and achieved a conductivity of more than 10^4 S·cm^{-1}, which is comparable with the conductivity of lead at room temperature (4.8×10^4 S·cm^{-1}). With the achievement of such high conductivity, CPs became a promising candidate material.

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Several studies have demonstrated the use of activated charcoal as reinforcement in polymeric composites. For instance, a report by Rajini *et al*., (2021), activated charcoal from doum palm was used as a dopant in low-density polyethylene (LDPE) to improve its electrical conductivity. The results showed that the electrical conductivity of the LDPE increased with the addition of activated charcoal, with a maximum value of 3.76 S/m achieved at 3% weight fraction of activated charcoal. Similarly, in a study by Ali *et al*., (2021) activated charcoal produced from date palm pits was added to high-density polyethylene (HDPE) to enhance its thermal conductivity. The results showed that the thermal conductivity of HDPE increased with the addition of activated charcoal, with a maximum value of 0.73 W/mK achieved at 20% weight fraction of activated charcoal. The objective of this work is to evaluate the electrical and thermal conductivity of the doped polymeric wastes using conductivity measurement techniques.

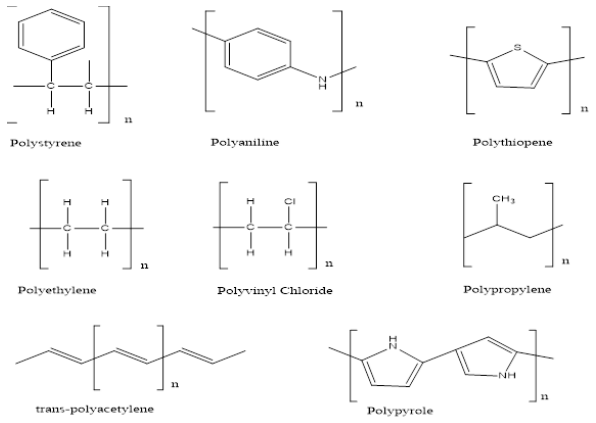

Figure 1: Structures of some conducting polymers.

2. MATERIALS AND METHODS

2.1 *Sample Collection and Pretreatment*

The samples used were sourced from some restaurants in Katsina metropolis, waste dump sites, road sites, and Yar'kutungu market in Katsina metropolis, Katsina State, Nigeria. Sachet water bags and water bottles were used as polyethylene source, Plastic and furniture housewares were used as polypropylene, White brittle pack used for protecting electronic gadgets and food packaging material were used as polystyrene, Activated Charcoal is obtained from burnt Doum palm bark. After the collection, the samples were taken to the Laboratory for identification.

2.2 *Preparation of Activated Charcoal*

Chemical activation was used in the preparation of the activated charcoal using chemical agents H_3PO_4 and KOH. The Doum palm was washed with water and oven-dried for 24 hours at 110ºC. The dried doum palm was ground, put in crucibles and introduced into the furnace at 600ºC in the absence of air or limited presence of Oxygen for 1 hour. The carbonized materials were recovered after 1 hour of carbonization and ground using a laboratory pestle and mortar and sieved until uniform granulated particles of 1 mm size. The small surface area has greater contact and hence stronger interfacial adhesion. The activation of the charcoal was realized using H_3PO_4 and KOH with a ratio of 5ml per 25g, and a contact time of 24 hours. Finally, the slurry obtained was washed with distilled deionized water until a neutral solution (pH of 7). Then filtered and dried at 100ºC in an oven for 2 hours. The powder obtained was kept in a clean container (Zhao *et al*., 2019).

2.3 *Method for the Doping of Polymers*

20g of the tiny cut pieces of each polymer was weighed into 250 ml beaker and heated at a regulated temperature of 130 ºC – 350 ºC to melt. The dopant of known different percentage concentrations 2.5, 5.0, 7.5, 10.0 in grams were prepared (Table 1). 25ml of each prepared concentrations were added into the beaker containing the polymer. The mixture was then stirred continuously to obtain a uniform temperature and a uniform distribution of the activated charcoal in the polymer matrix. The mixture was then poured into the mold, allowed to cool, solidified and compressed to form the composite tablet of the polymers (Thomas and Windle, 2008).

Table 1: Formulation of the doped polymers.

Weight of Polymers (g)	wt. % concentration of dopants (g)
20.0	0.00
20.0	2.50
20.0	5.00
20.0	7.50
20.0	10.0

2.4 *Characterization of the Activated Charcoal and Doped Polymers*

Characterizations were carried out using Philips XL30 Scanning Electron Microscope, VERTEX 70/70v spectrometer, Physico-Chemical Analysis, 500mega ohms multimeter 005-1349, TLS-100 thermal conductivity meter.

2.5 *Scanning Electron Microscope (SEM) Analysis*

The samples were examined under the scanning electron microscope (SEM) to analyze the morphological and surface characteristics. The SEM was carried out using a Philips XL30 Scanning Electron Microscope voltage of 10kV, beam size 3.0 working distance 2µm and magnification of 8000. The samples were coated with carbon under vacuum before analysis to prevent the accumulation of static electric charge on the surface of the samples. The microscope images of the samples before and after doping were taken.

2.6 *Fourier Transformation infrared microscopy (FTIR) Analysis*

Fourier Transformation Infrared FT-IR Spectroscopy was done using VERTEX 70/70v spectrometer (Agilent Technologies with spectra 4000cm-1 – 400cm-1). Scans were collected at the required cm-1 resolution to reveal the functional groups of the samples before and after doping.

Activation Efficiency Method

The activation efficiency R (%) is defined as the mass ratio of activated charcoal produced after activation to the mass of the sample used before activation. The yield value of the activated charcoal product is estimated by equation:

$$R(\%) = \frac{\text{Activated charcoal mass}}{\text{Precursor mass}} \times 100 \quad \cdots\cdots\cdots \quad (2.1)$$

Ash Content

The ash content C(%) was determined according to ASTM. In a dry crucible of mass m_0, 1g of activated charcoal is placed and weighed m_1. The crucible is then placed in a muffle furnace at 650^{0}C until the ash is obtained. After cooling it is weighed as m_2. The ash content is calculated from the formula:

$$R(\%) = \frac{m_2 - m_0}{m_1 - m_0} \times 100 \quad \cdots\cdots\cdots \quad (2.2)$$

Where:
m_0 = Mass of the dry Crucible
m_1 = Mass of dry Crucible + Mass of Activated Charcoal
m_2 =Mass of dry Crucible + Mass of the Ash

2.7 Measurement of Electrical Conductivity

The electrical conductivity of the samples was carried out by using 500mega ohms MASTECH multimeter 005-1349. Each prepared sample was tested with the equipment by placing it between the two opposite rods of the multimeter and its resistance was taken. Resistivity is the opposition given to flow of current per unit length of material of uniform sectional area and the reciprocal of resistivity will be measured as:

$$k = \frac{I}{R} \quad \cdots\cdots\cdots \quad (2.3)$$

Where k = Electrical conductivity R = Resistance

2.8 Measurement of Thermal Conductivity

The thermal conductivity was carried out by the use of TLS-100 thermal conductivity meter. The thermal conductivity was determined directly from the relationship between heat flow of the tested material and the temperature difference applied across. The results were obtained from the equation below.

$$\frac{Q}{t} = \frac{kAdT}{dx} \quad \cdots\cdots\cdots\cdots (2.4)$$

Where Q = heat flux (w/m^2), dt/dx = temperature gradient (k/m), A= cross section, t= change in time, and K= thermal conductivity constant.

3. RESULTS AND DISCUSSION

3.1 Scanning Electron Microscope (SEM)

The surface characteristics, morphology and micrographs were observed by SEM and the results are shown in Figure 2 for the activated charcoal before the doping and Figure 2(a, b and c) for doped polypropylene, polyethene and polystyrene respectively. All the polymers were in 10%wt dopant concentration in grams.

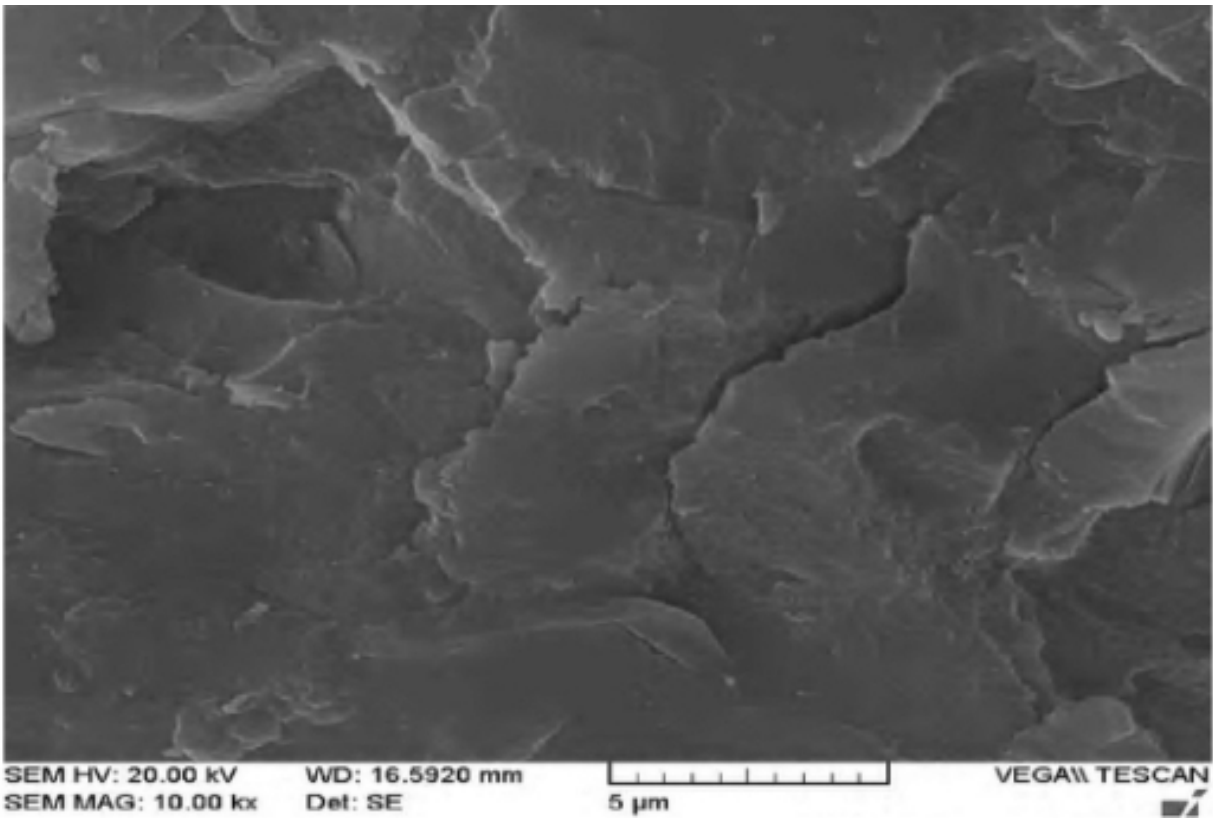

Figure 2: The SEM Image of the Activated Charcoal before the Doping

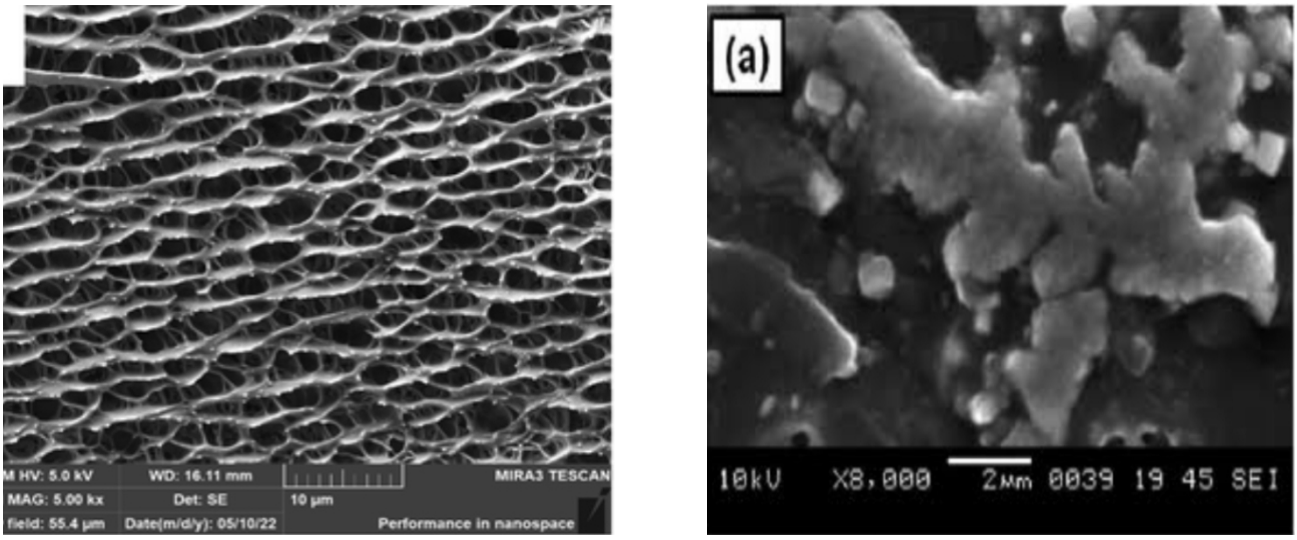

Figure 2a: SEM Image of Polypropylene before and after Doping

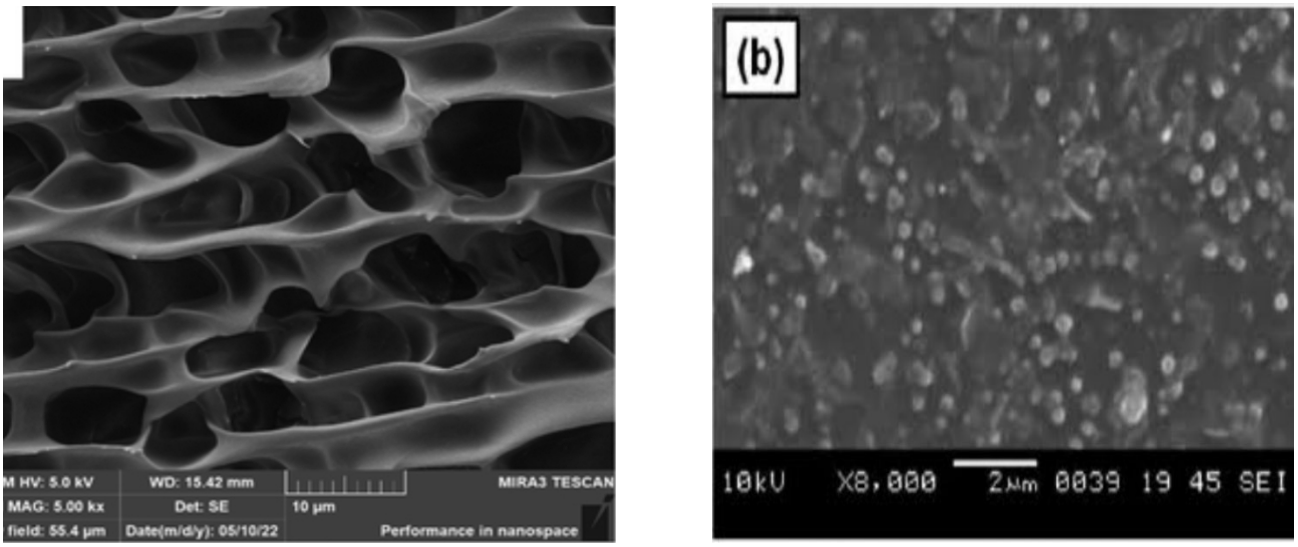

Figure 2b: SEM Image of Polyethylene before and after Doping

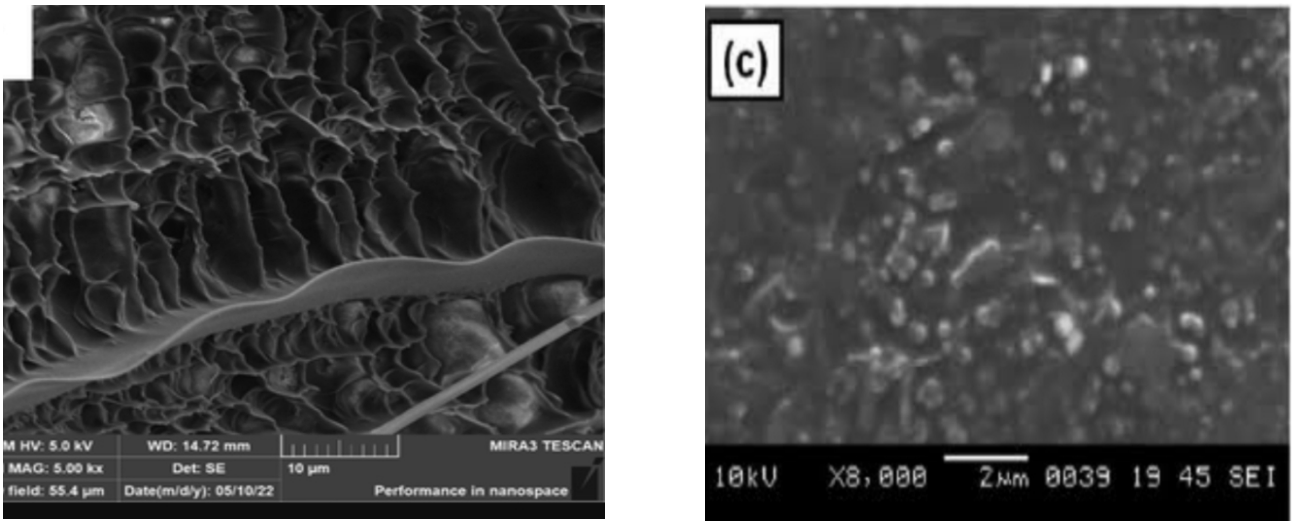

Figure 2c: SEM Image of Polystyrene before and after Doping

The SEM images of these pure AC and Polymers showed little heterogeneous morphology of the surface with small pores and cavities. High porosity and irregularity can be seen with the samples after the doping. The AC particles were found to be evenly distributed throughout the polymer matrix more especially in Figures 2b and 2c. The increase in the porosity and cavities for these doped polymers results in creating a network structure that allows for better electron and heat transfer which is believed to be responsible for the improvement in the electrical and thermal conductivity of the doped polymers. These changes were in good agreement with the literature reported for various polymer materials (Hassan *et al.* 2019).

3.2 *Fourier Transform Infrared (FTIR) Spectroscopy*

The FTIR analysis of the samples was carried out between 4000 cm-1 – 400 cm-1. The peaks appeared on the spectrum were assigned to various functional groups according to their wave numbers. Figure 3 a, b and c below shows the FTIR spectra for the doped polypropylene, polyethylene and polystyrene at 10% wt dopant concentration in grams respectively.

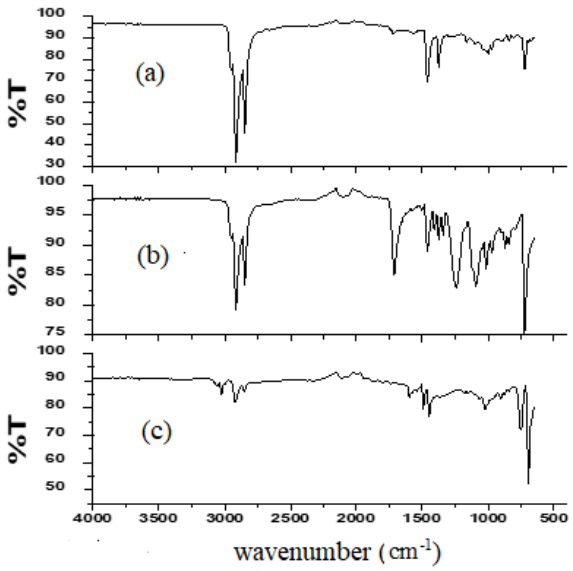

Figure 3: (a, b and c)- The FTIR analysis of the doped PP, PE and PS

The FTIR analysis of the doped polymers was carried out between 4000-400 cm^{-1} to determine the vibrational frequency changes of the functional groups in the samples. Figure 3a (doped polypropylene) the bands observed around 2914, 1722, and 1461 cm^{-1} are related to methylene C-H stretching, COOH acid and C-H bending. The rest at 998 and 723 cm^{-1} confirmed the presence of C=C bending. From Figure 2b (doped polyethylene) the peaks at around 2914 and 1714 indicate the presence of methylene C-H asymmetric stretching and COOH acid. The one at 1095 cm^{-1} is assigned to C-N stretching as it falls within the frequency range of 1250-1020 cm^{-1}. The presence of C=C bending was confirmed as shown in the peaks of 872 and 723 cm^{-1}. Lastly, for the (doped polystyrene) Figure 3c the bands that appear around 2922 and 1870 cm^{-1} can be assigned to methylene, C-H asymmetric stretching and C-H bending aromatic ring. The other ones at around 1595 and 1155 cm^{-1} are assigned to C=C stretching and amine CN stretch respectively. The rest at 752 and 693 cm^{-1} confirmed the presence of C=C bending as they all fall within the frequency range of 1000-650 cm^{-1}. These FTIR results were concordant with the results obtained by Adhikari *et al.* (2017).

3.3 *Physico-Chemical Analysis of the AC: Activation (yield) Efficiency and Ash Content*

Table 2 shows activation efficiency and ash content with two different chemical agents. CAA indicates the Charcoal Activation with acid while, CAB indicates the Charcoal Activation with base.

Table 2: Physico-chemical parameters of the Prepared Activated Charcoal.

Activated Charcoal	Activation Efficiency %	Ash Content
CAA	40.47	7.00
CAB	24.86	12.60

From the table above, it can be seen that the activation efficiencies of the prepared activated charcoal are less than 50% indicated the formation of microspore and mesopore. In the two cases of activation, the good mass yield obtained with the acidic agent H_3PO_4 (40.47%) and the weak mass yield obtained with KOH (24.86%). This could be explained by the combustion reactions that are very pronounced with H_3PO_4 because of its low stability temperature (boiling at 213 °C). While the base KOH is a strong base, corrosive, and stable product that can melt at 318°C. Similar observations were reported by (Haimour and Emeisch, 2006). Since the yield is an indicator of the performance of the preparation method, it can be concluded that H_3PO_4 activating agent have major advantage because of its good yield.

One of the parameters also influencing the quality of activated charcoal is its ash content. It appears that high ash content decreases the surface area. Therefore, ash content of the good, activated charcoal must not be too high, i.e less than 20% (Ayvral, 2009). The study shows the activation with Phosphoric acid shows low ash content compared to that of KOH. This could be assigned to the corrosive action of Phosphoric acid which eliminates impurities on precursors (Gueu, 2007). The nature of the chemical agent, therefore, has a considerable influence on the ash content.

3.4 *Electrical Conductivity on Doping with Charcoal*

The result of electrical conductivity of the samples is indicated in Figure 4. It was observed that the conductivity had increased linearly by the addition of dopant at different degrees.

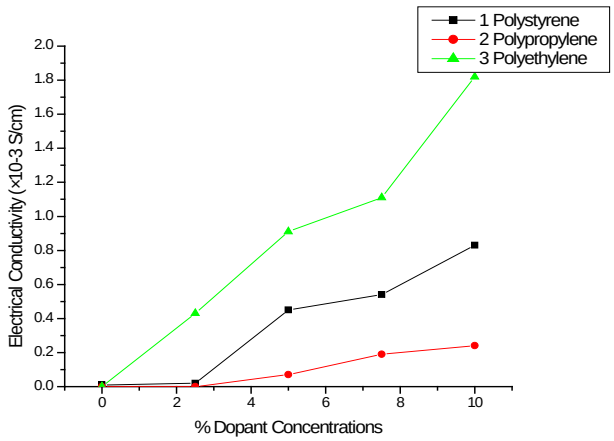

Figure 4: Effect of Dopant on Electrical Conductivities of the Polymers.

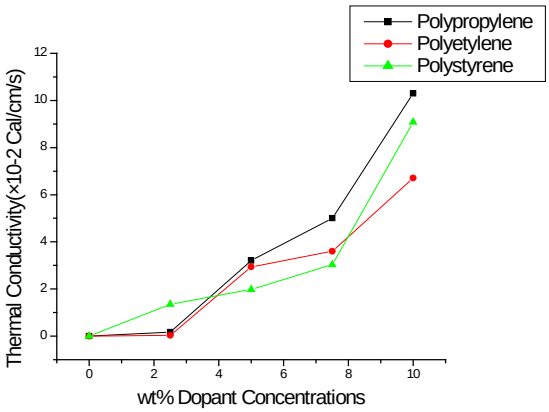

Figure 5: Effect of Dopant on Thermal Conductivities of the Polymers.

This arises from the thermal motion of the ions cores which causes lowering in the resistance character. Addition of electron donors or acceptors causes doping that result in electrical and magnetic changes in the inherently conducting polymers along with increasing conductivity or approaching the metallic range (Srilalitha et al., 2013).

Charcoal is amorphous in nature and its imprisoned electrons in the hexagonal carbon arrangement are free for movement (Seymour and Chery, 2008). This could account for better electrical conductivity in these polymers used because there will be i. More carrier electrons available, more space for hopping in the disordered arrangement of amorphous charcoal. ii. The conductive band of the carbons of the charcoal are brought closer to the valence band of the polymers for movements of electrons and holes.

The increment in the electrical conductivity of the doped polymers may be due to the order of amorphousness and also with the increased in dopant concentrations. The increment was most marked with Polyethylene being the most amorphous with short branches and followed the same trend to Polystyrene with dipole nature and the most crystalline Polypropylene with the least electrical conductivity (Chen et al., 2021). The optimum electrical conductivities were 0.24, 0.83 and 1.82×10-3 S/cm for PP, PS and PE at highest dopant concentrations of 10%wt. This is in line with the study by Ulenya et al., (2016) and Li et al., (2017) which used 5g each of the polymers and dopant from 0.00 to 1.5 wt% concentrations. The result shows the highest electrical conductivity at the highest concentrations of the dopant. The ranges of values were 0.16, 1.1, 0.9 and 0.79 S/cm for polypropylene, polyethylene, polystyrene and nylon 66 respectively.

3.5 Thermal Conductivity on Doping with Charcoal

Figure 5 showed the thermal conductivities of the samples at different percentages of dopant concentrations. It was revealed that polypropylene had the maximum thermal conductivity followed by polystyrene.

The ranges of values for thermal conductivity were 6.7, 9.08 and 10.3 ×10-2 Cal/cm/s for PE, PS and PP at the maximum concentrations of the charcoal dopant. The thermal conductivity of Polypropylene was observed to be highest followed by Polystyrene. There was spiking in polystyrene which resulted from increased thermal conductivity at lower concentration. This which could be attributed to the increased organization or compactness provided by the increased concentration of activated charcoal, which now increases heat transfer via photons. It can be explained that the lower concentration spike is accounted for by electron movements while the higher concentration spike is accounted for by elastic wave-like transfer of heat (photons) through the increased compact structure provided by the activated charcoal and direct transfer of energy from one atom to another (Ogochukwu, 2016). From the result, we can see that the addition of dopants increased the electrical and thermal conductivities of the polymers although the rate of enhancement is dependent on the intrinsic properties of the individual polymers. The most notable difference between conductive polymers and inorganic semiconductors is their mobility, and now recent advancements in molecular self-assembly are closing the gap which involves doping them. This is similar to the results reported by Batra et al. (2020) and Chen et al. (2021) in the study of doping strategies for the enhancement of electrical conductivity in polymer composites.

4. Conclusion

The results obtained in this research work have shown that the addition of dopants has significantly increased the electrical and thermal conductivities of the polymers although the rate of enhancement is dependent on the intrinsic properties of the individual polymers. The most notable difference between conductive polymers and inorganic semiconductors is their mobility, and now recent advancements in molecular self-assembly are closing the gap which involves doping them. Also, from the results it is evident that the level of increase in the electrical and thermal conductivities was found within the range (10^{-1} - 10^{-6} S/cm and 10^{-3} - 10^{-1} cal/cm/s) of the reported

conductive polymers, making them good and cheaper alternatives to be used in systems where such polymers are applied e.g. in production of capacitors, solar batteries, coating and potting of electronic components and the like. Furthermore, this research has uncovered the potential of using activated charcoal produced from Doum palm bark to improve the properties of polymeric waste, which can contribute to reducing the environmental impact of waste disposal while providing sustainable solutions.

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