

Evaluation Of Mechanical Properties and Microstructure of Heat-Treated Mild Steel Using Automotive Wastes as Energizer

K.K. IKPAMBESE¹, J.D. AMINE², M. ANYEBE³

^{1,2} Department of Mechanical Engineering, Joseph Sarwuan Tarka University Makurdi

³ Directorate of Road Traffic Services, Mabushi, Abuja

Abstract- Evaluation of mechanical properties and microstructure of mild steel treated using automotive wastes as energizer was carried out in this study. Automotive wastes (plastics, rubber, wood, fabric, non-ferrous metals, leather, glass, paper, textile, and dirt) were obtained and shredded, ground, sieved to sieve size of $\leq 300 \mu\text{m}$ and characterized. Seven (7) mild steel specimens cut from steel of 4 mm diameter were inserted into carburizing boxes using charcoal as carbon source with different percentage compositions of charcoal to automotive wastes (100:0, 90:10, 80:20, 70:30, 60:40 and 50: %50%). The same procedure was repeated by heat treating six specimens of mild steel without charcoal as carbon source by using different composition of automotive waste (0%, 10%, 20%, 30%, 40% and 50%) of the total volume of the carburizing boxes. The tests carried out to evaluate the mild steel specimens were hardness, impact strength, tensile strength, and microstructure examination. Results of characterization showed that the wastes contain different concentration of Iron, Nickel, Copper, Lead, Magnesium, Zinc, Manganese and Aluminium. Mechanical properties namely hardness, impact strength, tensile and percentage elongation were observed generally to improve with and without charcoal as carbon source. However, the level of enhancement was more pronounced with specimens treated with charcoal as carbon source. Hardness values increased with increasing percentage composition, as the highest hardness values of 295 at 10% of automotive waste and 90% of charcoal, while specimen with 50% composition of automotive waste alone gave the highest hardness of 172. The highest impact strength values of 91.73 J and 60.00 J were obtained with the 50% Charcol:50% automotive waste and 10% of automotive wastes without charcoal respectively. An

optimum tensile strength value of 660.35 N/mm² obtained at 70% Charcoal and 30% automotive waste. While tensile optimum value 5.314kN/m² was obtained with 40% of automotive waste in the absence of charcoal as carbon source. This will lead to cost reduction by avoiding the usage of additional alloying element. It is clear that automotive waste as an energizer can be used as substitute to conventional energizers in heat treatment of mild steel

Indexed Terms- Automotive wastes, Energizer, Mechanical properties, Microstructure, Mild steel,

I. INTRODUCTION

Ferrous metals and their components are usually opened to rapid wear and failure, this leads to significant increase in scheduled and unpleasant maintenance the costs in industries; thus leading to serious losses of functionality in a wide range of application. Increasing the mechanical properties of ferrous metals through microstructural modification will extend the life of ferrous metal parts thereby reducing both replacement and maintenance costs (Pahlevani *et al.*, 2016). Recently two major steps are employed in improving the microstructure and mechanical properties of ferrous metals; the first step is by increasing resistance of ferrous metals in their bulk solid form through microstructure modifications, using heat treatment. This is achieved through addition of alloying elements and secondly by surface engineering such as the application of coatings, films and surface treatments (Teixeira *et al.*, 2017). Consequently, there is significant industrial demand for further methods of surface modification to improve the mechanical properties of steel without affecting its bulk properties.

Energizers are substances that facilitate the reduction of carbon dioxide with carbon to form carbon monoxide and also release some elements that diffused to the surface of the treated mild steel. The formation of carbon monoxide is enhanced by energizers or catalysts, such as barium carbonate (BaCO_3), calcium carbonate (CaCO_3), potassium carbonate (K_2CO_3), and sodium carbonate (Na_2CO_3), which are present in the carburizing compound (Hosseini and Li, 2016).

Many researchers have utilized energizer in heat treating mild steel with the intentions of improving mechanical and microstructure properties. Ahaneku *et al.* (2012) studied the effects of heat treatment of mild steel in different quenchants using barium carbonate as energizer. The results of the tests showed positive changes in the strength properties of the mild steel material, in terms of high tensile strength, toughness, ductility and hardness. Thammachot *et al.* (2016) studied the effects of energizer, on the mechanical properties of hardened big knives of 70 mm x 30 mm in a pack carburizing process. Micro-Vickers hardness testing, impact testing and microstructure inspection were carried out and the results revealed that hardness derived from using CaCO_3 was slightly more than that using egg shells.

Sujita *et al.* (2018) investigated mechanical properties of pack carburized SS400 Steel using Pomacea Canaliculata Lamarck (PCL) Shell Powder as Energizer. The authors concluded that, PCL shell powder can replace the function of BaCO_3 and NaCO_3 as energizers on carburizing pack process. Automotive waste also known as automotive shredder residue (ASR) composed of plastic, rubber, wood, fabric, non-ferrous metals, leather, glass, paper, textiles and dirt. It is unfortunate that ASR with high potential of important alloying elements (carbon, nitrogen, silicon, aluminum and titanium) are usually found in dump sites

Pahlevani *et al.* (2016) investigated the possibility of modifying mild steel using automotive wastes without carbon source at different heat treatment times with single concentration of the waste. Metallographic investigation, mechanical properties results revealed that aluminium oxide formed on the steel surface in addition to silicone and titanium nitrides led to significant hardening and compressive strength. In this

present study, mechanical properties, and microstructure of mild steel treated with different concentrations of automotive wastes as energizer was investigated using charcoal as carbon source. Two set of experiments were conducted using the automotive wastes with Charcoal and automotive wastes without charcoal in the carburizing process.

II. MATERIALS AND METHODS

2.1 Preparation of the raw material

The automotive wastes (shown in Plate 1a) which include plastics, rubber, wood, fabric, non-ferrous metals, leather, glass, paper, textile, and dirt were obtained from Mechanic Village, North-bank, Makurdi. The wastes were shredded, ground (Plate 1b) and sieved using 300 μm sieve size. Charcoal from the wood was obtained from Wurukum Market and reduced to granules by using Pestle and mortar.



(a)



(b)

Plate 1: (a) Samples of automotive wastes collected and (b) ground and sieved automotive wastes

2.2 Characterization of the of the automotive wastes
 The automotive wastes were characterized using Atomic Absorption Spectrometer Perkin-El-mer model 460 to determine the elements present. A quantity of 20 g of the sample was measured and transferred into beakers according to the procedure in standard to form digested sample. The sample was introduced into the heating chamber of Perkin-El-mer model 460 spectrometer. Optics parameters and measurement parameters were adjusted for each of the elements detected as to measure the amount of energy absorbed by the samples. A detector was used to measure the wavelengths of light transmitted by the sample (the “after” wavelengths) and compared them to the wavelengths, which originally passed through the sample (the “before” wavelengths) as each atom has a distinct wavelength (Farshid *et al.*, 2016). A signal processor then integrates the changes in wavelength, which appear in the readout as peaks of energy on a computer attached to the equipment.

2.3 Surface modification using automotive wastes as Energiser

Mild steel samples of 4 mm in diameter cut into 20 pieces of 10 cm length each was utilized in heat treating. Heat treating mild steel using automotive wastes with and without Charcoal as carbon was done according to Farshid *et al.*, (2016). The samples were packed in a tight carburizing steel container (Boxes), in an automotive waste medium with enclosed granules of carbon powder of Charcoal. The percentage composition of the Charcoal powder and automotive waste were 50/50, 60/40, 70/30, 80/20, 90/10 and 100/0 % respectively. The furnace was initially pre heated at 200 °C, and the Carburizing boxes were introduced into the furnace. The temperature was set at 850 °C, and maintained for 2 hours (soaked) in an electric furnace. The temperature was increased to 1200 °C and the exposure to this high temperature environment in the hot zone at 60 minutes. The samples were brought back to

temperature of 250–300 °C for a further 15 minutes; once the allotted time had elapsed. This was done to prevent thermal cracking and the oxidation of the steel samples (Pahlevani *et al.*, 2016).

2.4 Hardness test

Two specimens of length 20 mm were cut from each of the modified steels with and without Charcoal using hacksaw. Prior to the hardness test, which used Vicker’s Microhardness tester (model: 900-391), the specimens were prepared for hardness test in accordance with ASTM E 92-17. A diamond point indenter of 0.5 mm diameter steel was used. The specimen was kept on the support table and the load is set to 100 kgf. The indenter touched on the specimen surface. After applying the load for minimum 15 seconds, the load release lever is pulled. The size of indentation (the diagonals) left by the indenter is measured. The larger the indent left by the indenter at a defined test force in the surface of a workpiece (specimen), the softer the tested material. The specimen is taken out and placed under microscope to view the reading on the dial. A total of three hardness values are taken on the work piece surfaces at three different locations. The results were recorded and average hardness value calculated.

2.5 Impact test

Two samples of lengths 55 mm obtained from the modified specimens from each of the compositions (with or without charcoal) were prepared using standard V-Notched Izod specimen specification in accordance with ASTM D 256 as reported by Chandl *et al.* (2016). The specimens after one another were clamped into pendulum impact fixture with the notched side facing the sticking edge of the pendulum. The pendulum was released and allowed to strike through the specimen and the impact or energy absorbed at the point of breakage for each of the specimens were noticed and recorded.

Table 1: Basic elements present in the automobile wastes by atomic absorption Spectroscopy

Element’s present	Iron (Fe)	Nickel (Ni)	Copper (Cu)	Lead (Pb)	Zinc (Zn)	Magnesium (Mg)	Manganese (Mn)	Aluminum (Al)	Others
Conc. (ppm)	0.0718	0.0057	0.009	0.2501	0.002	0.4785	0.0104	0.0081	0.1644

2.6 Tensile test

The tensile strengths of the specimens were measured with Testometric material testing machines. Prior to tensile test, the treated specimens were prepared according to the machine specification with length of 100 mm and diameter of 10 mm having a gauge length of 40 mm and in accordance to the BS standard. The test was conducted by gripping and pulling each specimen until catastrophic failure occurred. The load at which failure occurred was recorded. From each sample two specimens were tested and the average values of maximum tensile strength, and percent elongation at fracture were calculated (Narayan and Rajeshkannan, 2017).

2.7 Microstructural investigation

Microstructure examinations of the treated mild steel specimens were carried out using Digital metallurgical microscope (model: 254). Specimens with length of 20 mm were cut from the treated mild steel of different compositions of automotive wastes. Each specimen was carefully ground progressively on silicon carbide abrasive paper in decreasing coarseness (grain size). The ground surfaces were polished using gamma alumina powder on an emery cloth and the crystalline structure of the specimens were etched in 2% Nital solution

III. RESULTS AND DISCUSSION

3.1 Chemical composition of automotive wastes

Characterization of automotive wastes (Table 1) showed seven dominant elements in the waste. The elements found were Iron (Fe), Nickel (Ni), Copper (Cu), Lead (Pb), Magnesium (Mg), Zinc (Zn), Manganese (Mn) and Aluminium (Al). The result revealed the amounts of Fe, Ni, Cu, Pb, Mg, Zn, Mn and Al present were respectively 0.0718, 0.0057, 0.009, 0.2501, 0.0020, 0.4785, 0.0104 and 0.0081 ppm. These elements are capable of reacting with oxygen to form oxides of Aluminum, Zinc, Copper, etc. on the surfaces of treated mild steel thereby improving certain properties (corrosion resistance and Mechanical properties). Farshid *et al.* (2017) reported similar results in their study on surface modification of steel using automotive wastes as raw material. Similar elements found in their study were Iron (Fe), Nickel (Ni), Calcium(Ca), Copper (Cu), Lead (Pb),

Magnesium (Mg), Zinc (Zn), Manganese (Mn) and Aluminium (Al)

3.2 Effect of automotive wastes on Microstructure

Plate 1 (a and b) present the microstructure of untreated and treated specimens using 100 % charcoal as carbon source respectively, while Plate 1 (c-f) present microstructure of treated specimens using different composition of automotive waste. It was observed that there were microstructural changes associated with the carburization process of the mild steel samples treated using varying composition of automotive wastes as energizer with and without charcoal as carbon source; and this trend is in agreement with other Nwoke *et al.* (2014).The untreated steel microstructure (Plate 1a) shows a pearlite structure in a matrix of ferrite which is usually a lamellar combination of ferrite and cementite (Fe_3C), in contrast with microstructure of the treated samples which have dark phases of martensites in varying degrees of carbides that penetrated the surfaces as reported by Ihom *et al.* (2013). Carbon monoxide was produced as a reducing agent. This atomic and nascent carbon was taken by the steel surface, and subsequently it diffused towards the centre of steel sample. CO_2 thus formed react with the carbon (C) of the carburizing medium to produce CO, and thus, the cycle of the reaction continues.

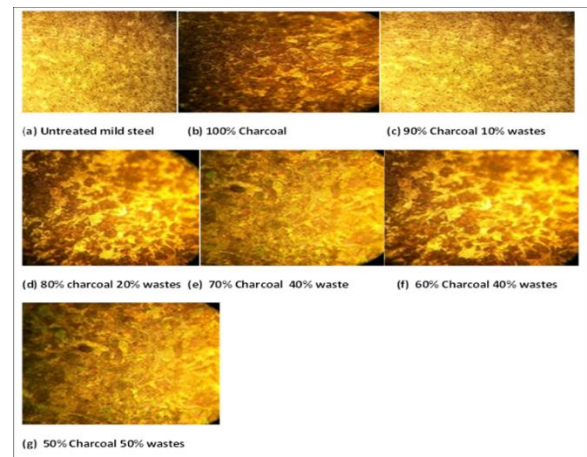


Plate 1: Microstructure of Untreated and Treated mild Steels using Different Compositions of Charcoal and Automotive Wastes

3.3 Effect of automotive wastes Composition on Mechanical Properties

3.3.1 Effect of automotive waste composition on hardness of treated steel specimen

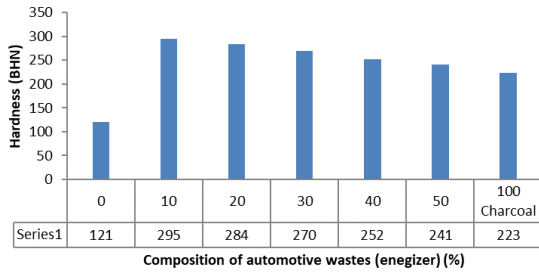
Figure 1(a) presents the effect of hardness values of steel samples treated with varying compositions of automotive wastes in charcoal as carbon source. There was improvement in the hardness values of the sample with the addition of automotive waste with the highest hardness value of 295 BHN at 10% of automotive waste using 90% of charcoal as carbon source. This trend was in agreement of other researchers such as Ihom *et al.* (2013) and Sujita *et al.* (2018) where egg shells and *Pomacea canaliculata* Lamarck shell powder were used in heat treating mild steel. The other hardness values obtained were 284, 270, 252, and 241 at 20, 30, 40, and 50% respectively. These values were better than the values of 121 and 223 for untreated (0%) and treated with 100% charcoal as presented in Figure 1(a) respectively. The results have shown that automotive waste is an energizer in the carburization of mild steel as the presence calcium and manganese that are capable of replacing use of carbonates (calcium carbonate, barium carbonate, and sodium carbonate) as energizers in carburization process. The values of hardness (295, 284, and 270) obtained were better than ones (52.6 and 262.3) reported by Ihom *et al.* (2013) and Suital *et al.* (2018). This implies that automotive powder as an energizer has aided faster diffusion of carbon into the material thereby reacting with calcium and manganese that are responsible for increasing hardness. This clearly shows imperative existence of the tertiary phase called martensite that is smaller volume of ferrite/pearlite that gave rise to increase in hardness. This also implies that samples obtained have the ability to resist plastic deformation under indentation when in use as agricultural implements.

The result of the Vickers hardness values of the treated mild steel using different compositions of automotive wastes without charcoal as carbon source is presented in Figure 1(b). It was obviously seen that hardness values increased with increasing percentage composition of the automotive wastes. Handoko *et al.* (2018) explained that the improvement in overall hardness is normally due to two reasons; firstly, the presence of elements such as manganese, and phosphorus, that manganese tends to increase hardenability of steel, but to a lesser extent than carbon. It is also able to decrease the critical cooling

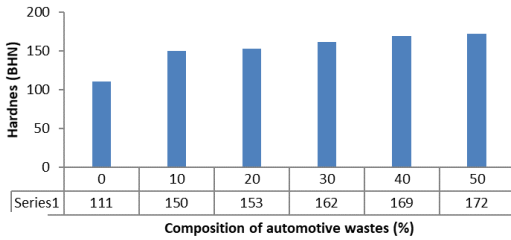
rate during hardening, thus increasing the steels hardenability much more efficient than any other alloying elements.

Secondly, the imperative existence of the tertiary phase called martensite that is smaller volume of ferrite/pearlite give rise to increase in hardness. The hardness values obtained were 121, 150, 153, 162, 169, and 172 at 0, 10, 20, 30, 40, and 50 % of automotive wastes respectively. The result implies that the percentage composition of automotive wastes affected the hardness values of mild steel. The values of hardness obtained were similar to the values obtained by Handoko *et al.* (2018) in their investigation on the improvement of corrosion resistance and hardness properties of carbon steel.

The optimum hardness value of 172 was obtained at waste concentration of 50%. This implies that at this level of concentration the specimen has more ability to resist permanent indentation when in contact with an indenter under load. It also means that the sample will be more resistant to scratching, cutting or abrasion due higher volume fractions in ferrite/pearlite phase (Handoko *et al.* 2018). That the main reason of this occurrence was because of compacted body-centered tetragonal (BCT) structure that promotes shear deformations which tends to generate tremendous group of dislocations known as highly stressed structure. The authors stated further that bainite is created from Fe_3C and dislocation-rich ferrite can forms intermediate hardness between martensite and pearlite phases. Pearlite is built from mostly ferrite phase with low percentage of Fe_3C that forms lamellar network relatively high in hardness properties than ferrite. However, the stress relief that included the recovery of dislocations will lead to the reduction in hardness properties at lower-level of quenching rate. In summary, the hardest microstructural phase was martensite, followed by bainite, pearlite, and ferrite.



(a) With charcoal



(b) Without charcoal

Figure 1: Effect of Automotive Wastes Percentage on Harness of Treated Specimen

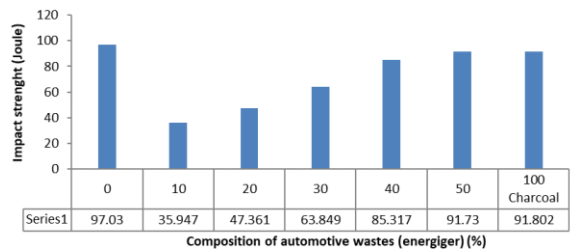
3.3.2 Effect of automotive wastes composition on impact strength of treated steel specimens

The effect of automotive waste (energizer) composition on impact strength of mild steel treated with charcoal as carbon source is presented in Figure 2 (a). It was observed that there was a sharp decrease in impact strength from untreated mild steel from 97.03J to 35.947J at 10% composition of automotive waste. Thereafter there was increase in impact strength as the composition of automotive wastes increases; the values obtained were 35.947, 47.361, 63.849, and 91.73J at 10, 20, 30, 40 and 50% respectively. The decrease in impact strength from untreated to treated samples (with % variation automotive waste and 100% carbon) could be the absence of pearlite, spheroidized cementite, tempered martensite and transformed retained austenite which play very important role in the improvement of impact toughness as reported by Chandl *et al.* (2016). However, these values were less than the impact strength value of 91.802 for sample treated with 100% charcoal. The decreased in absorbed energy is due to the formation to the martensite structure which is brittle and hard; as magnesium is found to decrease ductility leading to decrease of impact strength, as reported by Kadhim (2016). Nwoke *et al.* (2014) also reported that the general reduction in energy absorption was as a result

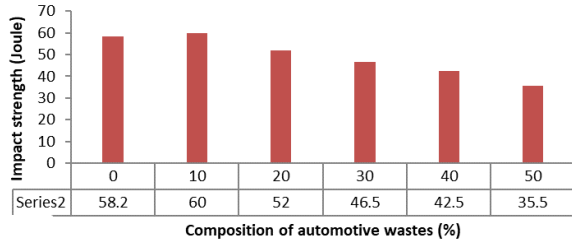
of increased diffusion rate and saturation of carbon atoms in the alloy thereby causing strengthening.

The result of the impact strength as presented in Figure 2(b) for the treated specimens using different percentages of automotive wastes without charcoal was observed to decrease with increasing compositions of wastes. However, there was slight increase of impact value from 0% to 10% waste composition before nose diving to 20%. This trend was similar to the one reported by Nwoke *et al.* (2014) in their investigation on the effect of process variables on the mechanical properties of surface hardened mild steel. This variation of impact energies is also attributed to the presence of course pearlite (annealing), fine pearlite and small amount of martensite (normalizing) and tempered martensite as a result of magnesium in the wastes (Chandl *et al.*, 2016).

The lowest impact value of 35.50 J was obtained at 50% of automotive wastes and the highest impact value of 60.00 J was obtained at 10% composition of automotive wastes. The highest value of 60.00J at 10% automotive waste composition is due to the tempered martensite and transformed retained austenite in the matrix of ferrite in case of mild steel specimen investigated as reported by Chandl *et al.* (2016). Other impact strength values were 58.8, 42.30, 46.50, and 52.00 J at 0%, 40%, 30%, and 20% compositions of automotive wastes. The implication of the impact strength value of 60 J at 10% composition of automotive wastes is the specimen can be able to withstand a reasonable energy before fracturing under a high rate of deformation compared with other specimens investigated.



(a) With charcoal



(b) Without charcoal

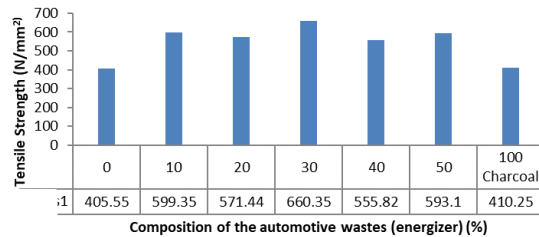
Figure 2: Effect of Automotive Wastes Percentage on Impact Strength of Treated Specimen

3.3.3 Effect of automotive waste composition on tensile strength of treated steel specimens

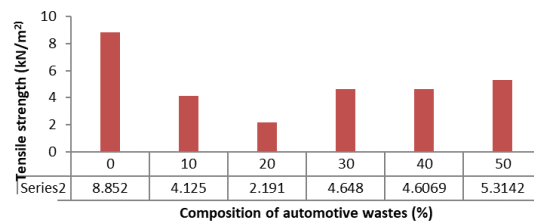
Figure 3(a) presents the effect of automotive waste (energizer) composition on tensile strength of steel treated with Charcoal as Carbon Source. It was generally observed that there was increase in tensile strength as the percentages of the automotive wastes were increased. However, the trend of increase was not in particular order, as the untreated mild steel (0%) and treated mild steel with 100% charcoal gave tensile strength values of 405.55 and 410.25 N/mm² respectively. These values were less than the values of 599.35, 571.44, 660.35, 555.82, and 593.1 N/mm² at 10, 20, 30, 40, and 50% respectively for automotive waste composition. It was observed that the best improvement in tensile strengths of 660.35 N/mm² was obtained with 30% automotive waste and 70% charcoal. The increase in tensile strength can be attributed to the higher volume fraction of the harder martensite which invariably decreases the ductility as reported by Ununakwe *et al.* (2017) in their study on effects of carburization with palm kernel shell/coconut shell mixture on the tensile properties and case hardness of low carbon steel.

Alaneme *et al.* (2017) also affirmed that the low tensile strength of the treated specimens of mild steel can be attributed to the fine lath martensite structure. The authors further stated that edges of the needle like lath structure serve as sites for stress concentration and triaxial stress state in the microstructure. Stress concentration and triaxial stress state constrains yielding of the material and facilitates micro-crack nucleation and propagation which culminates in fracture even when the nominal applied stress on the material is relatively low.

The result of tensile strength without charcoal as presented in Figure 3(b) was observed to decrease with increasing percentage composition of automotive from 0% to 20% and suddenly increased from 30% to 50%. The decrease in tensile strength with increasing percentage compositions generally can be attributed to the higher volume fraction of the harder martensite which tends to reduce the ductility of the mild steel. The result is consistent with the effect of tempering, which is normally used to relive internal stress, reduce brittleness and hence, toughness to resist shock and fatigue as reported by Adamu *et al.* (2019) in their study on evaluation of combined heat treatment techniques of testing hardness and tensile strength of mild carbon steel commonly used in Nigeria. The tensile strength values were 8.852, 4.125, 2.286, 4.648, 4.6069, and 5.314 kN/m² at 0%, 10%, 20%, 30%, 40%, and 50% respectively. This implies that the automotive waste used does not contribute meaningfully in improving tensile properties of the mild steel, due to little or no amount of Copper. However, at 40 and 50% composition of automotive waste there was significant improvement in tensile strength values of 4.6069 and 5.314 kN/m² respectively compare to other percentage composition of automotive wastes utilized.



(a) With Charcoal



(b) Without Charcoal

Figure 3: Effect of Automotive Wastes Percentage on Tensile Strength of Treated Specimen

CONCLUSION

The following conclusions were arrived at the end of this study

1. The automotive wastes obtained contained elements such as Iron (Fe), Nickel (Ni), Copper (Cu), Lead (Pb), Magnesium (Mg), Zinc (Zn), Manganese (Mn) and Aluminium (Al) in concentrations of 0.0718, 0.0057, 0.009, 0.2501, 0.0020, 0.4785, 0.0104 and 0.0081 ppm respectively. These elements are capable of reacting with oxygen during heat treatment process to form oxides of Aluminum, Zinc, Copper, thereby enhancing mechanical properties and corrosion resistance.
2. Mild steel treated with various percentage compositions of automotive wastes in the presence and absence of charcoal as carbon source showed improvements in microstructure which in turn affected hardness and impact strength potentially. However, improvement in microstructure, and mechanical properties were higher for specimens treated using charcoal as carbon source. It is clear those automotive wastes as an energizer, can be used as a substitute for conventional energizers in heat treatment of mild steel.

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