Development of Silver from Used X-Ray Films and Graphene from Drained Cassava Starch

Oluwafemi Samuel Dada
Department of Mechanical Engineering, College of Engineering and Technology, Kwara State University, Malete, P.M.B 1530, Ilorin, Kwara State, Nigeria

Seyi Rachel Dada
Department of Civil Engineering and Architecture, Tallinn University of Technology, Estonia

Jamiu Olaniyan Abdulhakeem
Department of Mechanical Engineering, College of Engineering and Technology, Kwara State University, Malete, P.M.B 1530, Ilorin, Kwara State, Nigeria

ABSTRACT:
This project discusses the development of silver and graphene from waste materials such as used X-ray films and drained cassava starch. This silver was extracted from X-ray film through a stripping process where all the elements were bleached off the film after which the sludge in the solution was allowed to settle before siphoning the water and finally heating the sludge. The graphene was developed from starch drained from cassava water, the cassava water was collected, sieved, and dried to collect the starch, after which Conc. H₂SO₄ was added to the starch to convert it to graphene. Hence, the SEM and EDS analysis of the extracted metals were carried out to determine the morphology, structure, and elemental composition. These analyses confirmed the extraction of silver and graphene from wastes as described above. Thus, these wastes were used to mitigate their extraction from their ore, which in turn would reduce exploration of its ore thereby conserving these raw materials.

Keywords: Silver extraction, graphene synthesis, waste materials, X-ray films, cassava starch.


INTRODUCTION
One of the major problems of the world especially in Nigeria is the improper disposal of waste [1]. Every day in life waste materials ranging from household wastes to industrial residues, cause detrimental effects on the environment and human health such as cholera, flood, pollution, and many more to mention a few. Wastes are trash but can be transformed into useful products for individuals and industries. Eradicating waste from our society seems impossible, but this trash can be recycled to reduce pollution that can arise from its presence in our environment [2,3]. Thus, wastes hurt our society. However, wastes can be exploited as utility resources to prepare wealthy products through recycling, other than burning, burying, using incinerators, and landfilling which is not an efficient way of control. Keeping the planet safe is important for our future generation to have a place to stay. It is interesting to make a new product from trash because recycling makes the environment clean, helps to conserve materials, saves energy, and reduces garbage in landfills [4].

Recycling wastes helps conserve raw materials and preserve natural habitats. Pyrolysis, a thermal decomposition technique, generates gases liquid phases, and solid char [5]. Waste plastics can be...
used as raw alcohols, while biomass waste can be converted into valuable chemicals like biofuels and alcohols. Automotive waste, like glass, can be used to prepare ferrosilicon alloys and silicon carbide nanoparticles. Moreover, ceramics with high mechanical strength and inertness can also be prepared from waste. For example, laboratory silica has been transformed into mullite by arc plasma method by [6], which is a well-known advanced ceramic with outstanding creep resistance and thermal shock resistance. Similarly, nuclear waste has been used to make glass and ceramics using an arc plasma method. As discussed earlier, recycling waste materials into high-value products such as steels and ceramics is markedly possible, which has inspired us as researchers to extract silver and carbon-based nanomaterials (CNMs) from waste resources for various applications [7]. Due to the high demand for precious metals, the study of their extraction from various forms of waste has increased. The waste produced by industries is of two components; organic and inorganic, organic wastes can be changed to a harmless substance before disposal. Inorganic waste comprises metallic and non-metallic components, from its metallic part precious metals such as silver can be obtained. Additionally, Silver is the highest electrically conductive element that has the highest thermal conductivity compared to other metals. This super precious metal is nearly used in all industries as it is useful to the photographic, electronic, automobile, food processing, and other industries [8]. Silver can be extracted from different waste which includes photographic films, camera film, X-ray film, and a scrap of a plasma TV. Photographic film waste contains 1.5 - 2 % (w/w) black metallic silver, 18 - 20% of the world's silver needs are supplied by recycling photographic waste. It is worthy of note to know that electronic devices such as switches, and superconductors are made from silver. Silver ions can be used for killing bacteria like fungi and algae. Also, this element helps to purify, sanitize, and filter water.

On the other hand, graphite is a solid and, a good conductor of heat and electricity. It is being used in the aviation, steel, plastics, and automotive industries. This material is an excellent conductor of heat and electricity, it's also corrosion-resistant. However, it has been found useful for producing different things such as lead pencils, lubricants, and crucibles and as a nuclear reactor. In nanotechnology CNMs, such as graphene and carbon nanotubes (CNTs), have been materials of priority for research because of their robust mechanical, electronic, physical, and chemical properties are very good in super-capacitors, transistors, transparent photo-electronic devices, and sensors. A variety of synthesis methods have been used to produce CNMs [7]. High-quality CNMs can be produced through those conventional methods, but one critical challenge that we are trying to achieve is environmentally friendly production on a large scale with low cost. Synthesis of CNMs starting from wastes will reduce the number of solid wastes and lower the fabrication cost. More importantly, using waste as a resource is a good step for recycling technology. Wastes with high carbon content, especially waste plastics, are considered as pronounced precursors for making CNMs.

Again, the environment is supposed to be a place free from pollution: air, land, noise, and water pollution, but wastes have polluted society as a result of their improper disposal [1,9]. Although measures have been taken to eradicate this from our society, the government has created a method of waste disposal, that involves using trucks to pack wastes from homes and streets, but there has not been any great change because the environment remains polluted with various kinds of trash. This waste causes detrimental effects on the environment and human health such as cholera, flood, pollution, and many more. However, eradicating these wastes from our society seems impossible as burning, burying, using incinerators and landfilling are not an efficient way of waste control, but this trash can be recycled to reduce the effects that can arise from its presence in our environment.

Hence, the conservation of raw material is also a factor we intend to focus on that is, how to convert our waste to wealth. Raw materials are conserved and natural habitats are being preserved for the future as a result of recycling wastes, this is easy to do because less energy is needed for this operation as compared to creating a new product from raw materials. Keeping the planet safe is important, for our future generation to have a place to stay. This is essentially the reason why it is mandatory and expedient to make a new product from trash through recycling which in turn makes the environment clean, helps conserve materials, saves energy, and reduces garbage in the landfill. Thus, the next segment shall discuss the aim and objective of the work. Therefore, this project aims to develop silver and graphene from waste such as photographic films, x-ray films, and starch from cassava-drained water. The specific objectives of this project are:
• to produce silver from waste;
• to produce graphene from waste; and
• to characterize both the silver and graphene developed.

The rationale of this study is to reduce environmental pollution by recycling industrial and agricultural waste for the production of silver and graphene. This will serve as a source of revenue for the nation and help create employment opportunities for unemployed graduates. The human population has seen the final destination of waste to be in the bin but this is not true because these wastes can be transformed into a viable product for individuals and industries, converting this trash to an economic utility will help reduce pollution, health diseases caused by this waste, create job opportunities, and generate revenue for the nation.

**METHOD AND MATERIALS**

**Silver Extraction**

**Materials Used:**

*Buckets* were used to store water into which the chemicals were dissolved to complete the extraction process.

*Crucible or Melting Dish* was used to evaporate all the water left in the solution obtained after the extraction process was completed.

![Figure 1. Bucket used to Store Hot and Warm Water](image1)

![Figure 2. Crucible used to Evaporate the Water Left in the Sludge](image2)

*Digital scale* was used to weigh each chemical before using them.

*Used X-ray films*: The films were collected from a friend whose family members did a series of X-rays at UIITH Ilorin, and it weighed about 60gram so it was enough for our project.

*Sodium hydroxide*: This chemical was purchased at Ojota market in Lagos State. It is a white coloured chemical properties/characteristic

*Sodium Sulphide* this chemical was purchased at the Ojota market of Lagos State. It has a rotten egg smell and is white in colour.
Method of Extraction of Silver

Three (3) litres of water were boiled and poured into a transparent bucket, then 60 grams of Sodium Hydroxide powder was added into the hot water, after which the used X-ray films were cut into halves and each was dipped into the hot solution one after the other till they become clear. The process of bleaching the film is called the stripping process. After removing the X-ray films from the solution, the bleached films were dipped into clean tap water to rinse off the left-over Silver halides of the film. Then, 0.6 litres of Sodium Sulphide solution were poured into the sodium hydroxide solution. The Sodium Sulphide was used to precipitate the Silver solution. The new solution was stirred and covered overnight.

On the next day, a sludge was formed beneath the bucket, and the solution was tested to determine if all the Silver sludge had settled beneath the container before taking the next step. The testing was done by scooping a little of the solution into a small container-like cup then a pinch of the yellow Sodium Sulphide was pinched into the small container and stirred. After this operation was performed we waited for about 5-10 minutes. The bottom of the small container was checked for
black particles or sludge and none was found which connotes that there is no more Silver available in the solution but all had settled beneath to form sludge. The above process of testing the solution was performed also with tap water.

Thereafter, the solution was poured out slowly leaving the sludge beneath and this was poured into another bucket including the one from the tap water. The two buckets were rinsed with a small amount of water, and the water was poured back into the new bucket. This new bucket was covered and the sludge was allowed to settle for an hour, after which the water was poured out and the sludge was left beneath the container. The whole sludge was poured into a cloth bag and hung on a rope to drain off the water. Note that the first set of water that was drained was captured back into the bag since it still contained Silver. The sludge was poured into a crucible and was heated for the water to evaporate completely. This was done until the blue flame started showing.

**Characterization of Silver**

**Equipment**
- Scanning Electron Microscope (SEM)
- Energy Dispersive X-ray Spectrometer (EDS)
- XRD

**Method**
The microstructure homogeneity of the silver was checked using a Scanning Electron Microscope (SEM) analysis and Energy Dispersion X-ray Spectroscopy (EDS). It shows the weight, atomic mass, and error percentage of the element in the result obtained at different magnifications and different selected areas. It also tells the shell of each element. XRD analysis shows the presence of the required element in the extracted product to confirm that the required element is the one being produced as it's going to be the element having the highest peak in the composition.

- ZEISS Scanning Electron Microscopes (SEM) at AUST
- MiniFlex 300, X-ray Diffractometer (XRD) at ABU Zaria

**Graphene Extraction**

**Production of Starch from Waste Product**
The starch that was used in this study was obtained from cassava-drained water, concentrated sulphuric acid (Conc. H₂SO₄), a beaker, and a glass rod. Drain cassava water from processed cassava was collected in a bucket. The cassava water was left to settle, then the water at the top was poured away and the particles that settled down were collected. The particle collected is the starch needed. The starch was sundried to remove the remaining water in the starch.

**Graphene preparation**
20 grams of starch was poured into a beaker and the concentrated sulphuric acid (Conc. H₂SO₄) of 18ml was added into the starch, with a glass rod, the mixture of starch and the Conc. H₂SO₄ was stirred continuously. Then, the mixture formed was sun-dried.

**Characterization Methods**
Scanning electron microscopy (SEM), transmission electron microscopy (TEM), and high-resolution transmission electron microscopy (HRTEM) are usually employed to visualize the morphology and structure of graphene-based catalysts, which provides lots of information to rationalize their catalytic performance. The morphology and structure of the graphene extracted were visualized using a scanning electronic microscope (SEM) analysis and an energy dispersion X-ray spectrometer (EDS) was used to check the elemental composition of the graphene obtained from the extraction process, it tells the weight, atomic number and error percentage of the element in the result gotten at different magnification and different selected area. It also tells the shell of each element.
RESULTS AND DISCUSSION

SEM/EDX and XRD Investigations of Silver

Figure 8. shows the SEM images of silver extracted from X-ray films with a magnification of 200µm. Scanning electron microscopy (SEM) was employed to visualize the morphology of silver, which provides lots of information to rationalize their performance.
EDS was used to determine the elemental composition of silver Ag. The EDS spectrum confirmed that the extracted silver was composed of C, O, Na, Al, Si, S, Cl, Ag, Ca, and Fe. Moreover, the atomic percentages of species were achieved. Based on these results, they concluded that Ag was present in the composite sample. The EDS was also used to investigate the elemental composition.

Figure 9. Shows the EDS of the Silver Extracted from the X-ray Film

Figure 10. eZAF Smart Quant Results of the Silver, Showing the Various Constituents of the result.
SEM/EDX Investigations of Graphene

Figure 13 shows the SEM images of synthesized thin films of graphene prepared from starch with magnification of 100µm and 200µm respectively. Scanning electron microscopy (SEM) was employed to visualize the morphology and structure of graphene, which provides lots of information about the extracted metal. SEM of graphene demonstrated a 2D planar structure with a well-defined and interconnected porous network.

EDS was used to determine the elemental composition of graphene G. The EDS spectrum confirmed that the G was composed of C and O atoms and sulphur. Moreover, the atomic percentages of species were achieved. Based on these results, they concluded that G was present in the composite sample. The EDS was also used to investigate the elemental composition. EDS analysis confirmed that the elemental ratio of the synthesized hybrid nanoparticles was stoichiometric for the component of sulphur atoms while the percentage of reduced graphene oxide (carbon to oxygen) ratio was in proportion.
Figure 13. SEM images of graphene of magnification of 100µm and 200µm respectively

Figure 14. EDS Graph of the Graphene Extracted from Starch

Figure 15. eZAF Smart Quant Results of the Graphene, Showing the Various Constituents of the Result
XRD graph result denotes that at the peak around 18, there is the presence of θ Carbon sulfide (CS₂) and at peaks 27 and 56 degrees there is the presence of β Carbon (C), at 43 degrees there is the presence of Lonsdaleite (C) and at peak 62 degree there is a presence α Graphite (C) which are allotropes of graphene.

CONCLUSION AND RECOMMENDATIONS

Silver has been developed from photographic waste and graphene has been developed from cassava waste (starch), Scanning Electron Microscope (SEM) and Energy Dispersive X-ray Spectrometer (EDS) have been used to test the result of the silver and graphene developed. SEM analysis was used to check the morphology of silver and graphene, and EDX was used to check the
elemental composition of the silver and graphene developed. XRD analysis has been done to confirm the development of silver and graphene. In the analysis, we found out that silver and graphene had the highest peaks in their respective compositions. Since the development of silver and graphene has been done, and the main scope of this project is to combine the two materials using SiC or ZnBr, we recommend that this project should be continued and completed by combining the two materials using binders to form a composite material that is very light, even lighter than aluminum and with various good thermal, electrical, tensile strength, hardness, stiffness, and other desirable properties.

LIMITATIONS OF THE STUDY
The challenges faced in the course of the project are as follows:
1. It was difficult to get the waste, especially in the development of silver; we had to go from one hospital to another to get the photographic film.
2. There was no lab and necessary equipment in the department to experiment.
3. The research is costly
4. Sending samples for analysis is expensive and tedious, because we have to send them to Abuja for the SEM and EDS, and have to send the result to Zaria for XRD analysis.
5. Being the first time doing these analyses, it was difficult to understand the result from the analysis

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