The Scientific Temper
Vol. 12, No 1-2, January-July, 2021:pp 9-15
ISSN 0976 8653, E-ISSN 2231 6396
A Web of Science Journal
Doc ID: https://connectjournals.com/03960.2021.12.9

Biowaste-derived Nanoparticles and Their Preparation: A Review

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ABSTRACT

The global population is increasing drastically by which the consumption of natural resources is more and more, creating larger by-products which are left unused creating huge wastage. Different methods are to be incorporated to use these biowastes for human use. One of such alternatives is the preparation of nanoparticles using these biowastes.This process is called as green synthesis.Green synthesized nanoparticles have a wide application in both biomedical and physiochemical fields. These NPs (Nanoparticles) transition between bulk materials and atomic or molecular structures.The present review deals with the synthesis of green synthesized nanoparticles from various agricultural wastes, which alos include wastes from animals.

Keywords: Nanoparticles, Green Synthesis, Rice husk ash, Bovine bone powder, Eggshells, Fruit Peel Powder

INTRODUCTION:

The word 'Nano' comes from the Greek term Nanos, meaning a dwarf. More than thousands of products based on nanotechnologies are already being used in everyday life and many new nanoproducts are been expected to appear on the market within the next few years. Since past couple of decades, numerous products related to nanoparticles have been fabricated through various methods and are used in many advanced technologies.

There are different methods for nanoparticles synthesis like chemical assisted,sonochemical reduction,pulse laser method and green synthesis. Green synthsized nanoparticles have a wide applications which include nanosensing in crop protection for disease identification and growth stimulants and as capping and reducing agents.

Green synthesis is advantageous over the chemical counterpart method, as it is safe, simple, cost-effective, relatively reproducible, and often results in more stable materials (1,2). Due to the rich biodiversity of plants and their potential secondary metabolites, plants and plant parts have been well exploited in recent times in the synthesis of a variety of nanoparticles (3). Green synthesis is also thought to be responsible for binding biomolecules to the Metal nanoparticles surface during their synthesis. This phenomenon is commonly referred to as 'Capping' in the green synthesis field.

Agricultural waste is the waste produced as a result of various agricultural operations. It includes manure and other waste from farm poultry, houses and wastes from agricultural activities or the waste left when useful fruit or seed has been removed. There is tonnes of agrowaste produced every year. There are scientist constantly working on this field.

Studies have been conducted on agro material such as banana and orange peels (4), nutshells (5), maize cob, coconut husk fibers (6), bagasse pith, sawdustwastes (7), soybeans, and cottonseed hulls for their adsorptive properties. These materials have indeed been documented for adsorbing various pollutants such as heavy metal ions, dyestuff, and other harmful pollutants (8) and have been

evaluated for the adsorption of heavy metal ions from wastewater.

Studies on the use of agricultural by-products have included metal-binding studies with Daturainnoxia (9), oat fiber, dyed cellulosic materials, wheat and rice bran (10), sugarcane bagasse, maize cob, sawdust, etc. Agro-waste products are typically made up of lignin, cellulose as the major constituents. Hemicellulose, extractives, lipids, proteins, simple sugars, starches, water, hydrocarbons, ash etc. are minor components containing several functional groups present in the binding process. The mechanisms of binding of heavy metals by biosorption could be demonstrated by physical and chemical interactions amongst cell wall ligands and adsorbents by ion exchange, complexation, coordination, chelation, physical adsorption, and micro-precipitation process(11).Various agricultural waste have been used.

Rice husk is an agricultural waste produced in massive quantities of 100 million tons as a by-product of the rice milling industry, of which 96 percent is produced in developing countries. Polymerized onion skin with formaldehyde (12), Waste wool (13, 14), peanut skin (15), modified bark (16,17,18), Barley straw (19) have been studied for the removal of nickel by adsorption method (20). *Mangifera Indica* seed shell (21), coal-based absorbent (22), burned clay and root, furnace gas cleaning sludge (23), hydrous oxides of iron (24) have been reported.

Feathers constitute 5% to 7% of the body weight of chickens. These significant by-products of the poultry industry generate millions of tons annually worldwide (25, 26). Chicken feathers consist of feather fiber and quill which are made of keratin (about 90% by weight). Keratin biomaterials derived from human hair are effective in preventing bleeding and enhancing survival in several animal models of bleeding (27, 28). That being said, there are few records of chicken feather keratins for haemostatic use. The production of feather keratin for haemostatic applications is very attractive and important for the recycling of discarded feathers (29).

Preparation of Nanoparticles using different types of Agrowastes

1. Saw Dust

a) Preparation of Fe_2O_3/ sawdust nanocomposite

Sawdust (5.0 g;35-50 mesh) is added to 500ml of 0.50 M $FeCl_3$ solution and stirred for 2hr at room temperature. Then, 1M NH_3 solution was slowly added into the mixture for 2h under vigorous stirring. Then the mixture is heated at 100°C for another 2h. The resultant α-Fe_2O_3 /SD particles were rinsed with DI water and dried for several hours in an oven at 100 °C (30). The nanoparticles formed are stored in a sterilized plastic bottle.

2. Biosynthesis of silver nanoparticles using Panchakavya

Panchakavya is the mixture of Cow milk (3L), Cow curd (2L), ghee (1/2 kg), Cow urine (2L), Cow dung (2 kg), Tender coconut water (3L), Bananas (10), Crude jaggery(1 kg), Water (3L).4 mL of panchakavya was mixed with 96 mL of 1 mM $AgNO_3$ solution, and incubated for 8 hrs in a rotary shaker (180 rpm) at room temperature. The change in color of the reaction mixture is observed from milky white to dark brown. To separate the silver nanoparticle the reaction product is centrifuged at 13,000 rpm for 15 min and purified by redispersion of the pellet in autoclaved water and later resultant nanoparticles are stored (31).

3. Synthesis of Platinum Nanoparticles from Bovine-Bone Powder

The platinum chloride, $PtCl_4$ was used as precursor salt. In 50 mL of deionized water 16.75 mg of $PtCl_4$ was dissolved to make 0.001 M solution. 0.01 M 50 mL of $NaBH_4$ is prepared. The bovine femur is washed, cleaned, and immersed in a 0.01 M HCl solution. The bone is then cut into small pieces and finely ground and sieved with 150 mesh. 975 mg of bovine-bone powder was immersed in 50 mL of $PtCl_4$ for 30 seconds and then filtered. Later $NaBH_4$ is added and the solution is stirred for 30 min. The resultant solution is dried overnight at room temperature (32).

4. Synthesis of silica Nanoparticles From Rice Husk (RH) using Chemical method

Alkaline extraction is an effective and easy technique for extracting amorphous silica from agricultural waste. The process utilized the alkali extraction technique from RH to extract silica particles to remove metallic impurities.

In a water bath, RHA was initially treated with HCl for 4 hours at 75°C. The filtration was done by using distilled water and constantly washing until a neutral state was reached and dried at 110°C for 12 hours. The NaOH was used to prepare a constantly stirring solution of sodium silicate for 1 hour at 90°C. The silicate sodium solution was then reacted to ethanol, and a steady 10-minute water mix was added. The whole mixture has been titrated 3 M H_3PO_4 until gel formation is carried out. The product after centrifugation of yellowish gel was washed with distilled water to clear away residual sodium silicate and phosphate, followed by calcination to produce silica nanoparticles(33).

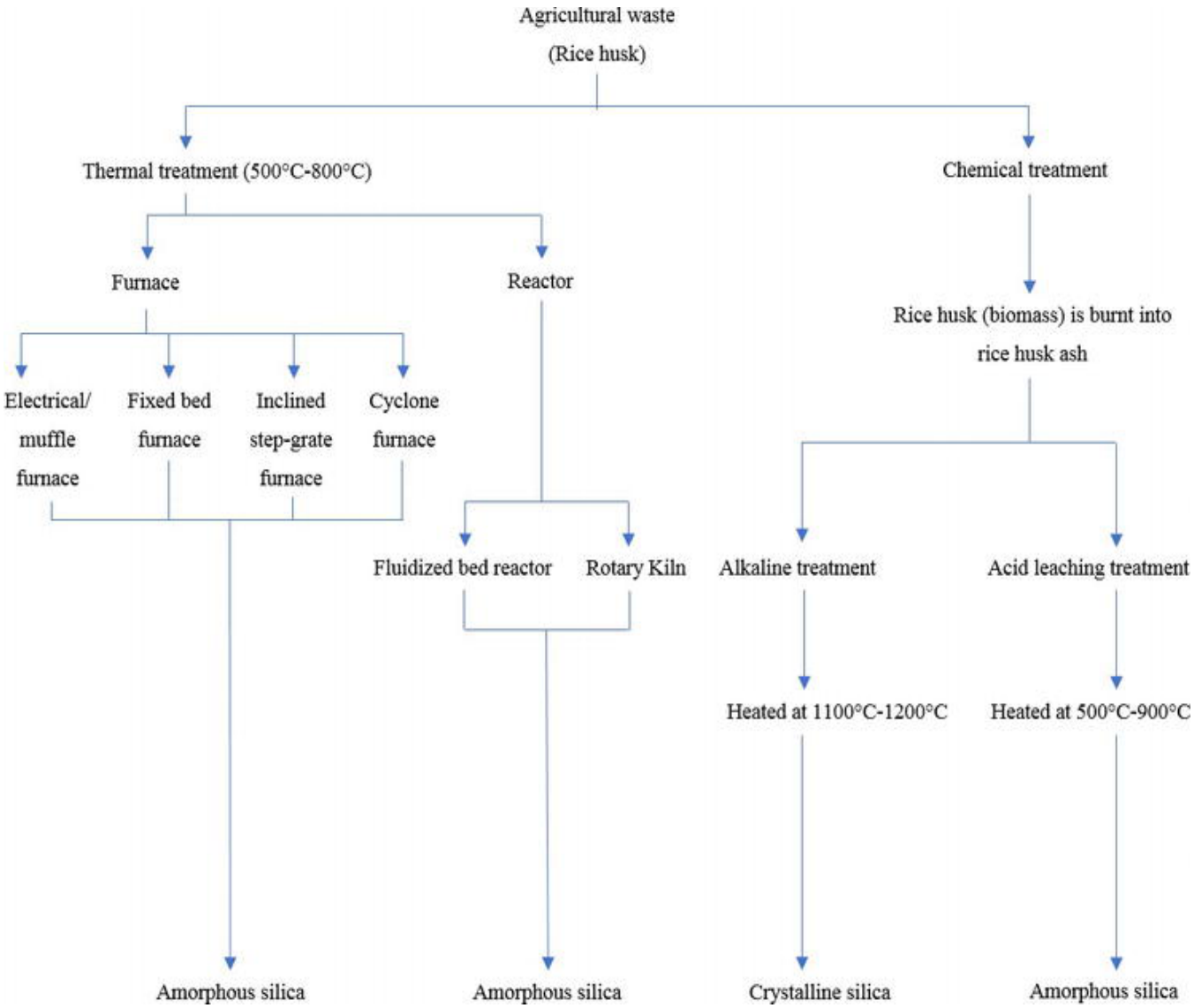

Figure 1. Various treatments used to produce nanosilica from agricultural waste.

5. Synthesis of Calcium Oxide Nanoparticles from Eggshells

Generally,the egg is in three parts: albumen, eggshell, and yolk with the components of water, lipids, proteins, minerals, and carbohydrates [1]. The chicken eggshell was make up of calcium carbonate and organic materials. Approximately the layer thickness is 315 µm.

Egg Shell Collection: Empty chicken eggshells were collected from household waste and washed under running water. The adhering membranes were manually removed. The eggshells samples were washed, cleaned, and sun-dried in the open air for 48 hrs followed by drying oven at 110 °C for 2 h. Dried and cleaned eggshells were then crushed and grounded in a blender to form eggshells powder. The resulting material was denoted by nanoCaO

Synthesis of Calcium Oxide Nanoparticle: The crushed eggshell powder was treated at above 700 ºC for 7 h. The gaseous state CO_2 escaped and later formed pure CaO (34).

6. Preparation of ZnONanoparticles employing goat intestine waste

Goat processing discard (principally gut) was collected and washed thoroughly with deionized water to get rid of dust or blood stains along with metallic contamination. The discards were immediately ground in the 50% ethanol, mixed, and gently heated until the colour of the mixture changes to deep straw. This extract was carefully filtered after gently meshing the animal tissues, pooled, and cooled for 20 min at room temperature. The finely meshed animal tissue was further boiled in a fresh pool of sterile

distilled water (200 ml) for another 10 minutes. This too was cooled and filtered to ensure complete extraction of the probable candidate metabolites. Thereafter, both the extracts were mixed and utilized for the preparation of any NPs (35).

7.Biosynthesis of ZnO nanoparticles Analytical reagent grade zinc chloride ($ZnCl_2$) was taken for and 0.25 (M) solution was prepared. 20 ml of solution was added to the diluted juice (without shifting the pH beyond 6) and it was heated until the appearance of starch-like haziness in solution and the white deposition at the bottom of the flask was noted. This was perceived as the initiation of transformation. The value of pH was maintained at 6 because a lower value delays the procedure of oxide synthesis and a higher value led to metallization. The flask was allowed to incubate for another 4 hours till white mass settles down leaving clear transparent supernatant at the top. The ZnO nanoparticle formed was filtered for further studies.

8. Biosynthesis of silver nanoparticles using Pine needles

Pine needles extract: For the synthesis of metallic NPs from pine needles, plant samples were collected, washed, and dried to form a powder. The powdered sample of pine needles was mixed with 250 mL double distilled water and boiled for 5 min. The heated materials were filtered and centrifuged to obtain a clear extract.

Synthesis of AgNPs: Silver nanoparticles were synthesized by adding 10 mL of 0.01 M aqueous solution of silver nitrate into different volumes of Pine needles extract taken into four beakers separately at room temperature. The colour of the solution was started changing from yellow to brown within 5 min indicating the formation of nanoparticles and further, no change in colour can be observed. The separation of silver nanoparticles from the dispersion was carried out by centrifugation after that AgNPs were washed 4 times with distilled water and acetone to remove water-soluble impurities and then nanoparticles were lyophilized and stored in dry bottles for further study(36).

9. Preparation of silver nanoparticlesfromthe fruit peel

Mixed fruit waste is collected from the houses and fruit markets. Then these fruits are separated from their peel and according to the fruit. After the segregation, these peels are washed under tap water first and distilled water later for serval times to remove impurities. Then they are dried at 105°C for 24 hrs in hot air over to dehydrate the peel.

About 250 ml 4 mM silver nitrate aqueous solution was prepared. 10 g of powdered fruit peel was transferred to a round bottom flask containing 250 ml of double-distilled water and then each was heated to boiled below 60°C for 30 min. 3 ml of fruit extract was carefully added to 40 ml of 1 mM aqueous $AgNO_3$ solution in a 250 ml flask and kept at room temperature for 5 hours under a dark chamber to minimize the photoreduction of silver nitrate solution at room temperature. A change in colour from colourless to reddish-brown or golden-brown of colloidal suspension confirmed the biosynthesis of silver nanoparticles. The solution is centrifuged at 3000 rpm for 4h, the pellet was oven-dried overnight. The formed nanoparticle is stored in an airtight container and used when required(37).

10. Preparation of silver nanoparticles fromFrom Weeds (*Cyperus rotundus*)

Preparation of extract: Fresh *C. rotundus* grass was collected, rinsed under sterile distilled water, and dried. After drying cut into small pieces. 20 g of *C. rotundus* grass was added in 100 mL of deionized water and then heated at 60 °C for 5 min. Later, the solution was decanted and filtered through nylon mesh. The filtrate is stored at 4ºC for further nanoparticles synthesis process.

Preparation of nanoparticles: The extract of 2ml was added in 25 ml of 1mM $AgNO_3$. The mixture was then stirred at 60°C for 30 mins. The change in the colour of the stock solution to brown within 20 min indicates the formation of silver oxide nanoparticles. The mixture was centrifuged and filtered. Then it is dried in Hot air oven at 80° C for 4hrs. The obtained powder is a silver nanoparticle (38).

11. Preparation of silver nanoparticles fromWheat straw

Preparation of the wheat straw solution: The wheat straw was sun-dried for 5 days, and grounded into a fine powder with the help of a pulverizer at 25,000 rpm for 15 min. Approximately 5 g of wheat straw powder was diluted in 200 mL of deionized water. The sample was ultrasonically mixed before standing for 10 min at room temperature. The supernatant solution was filtered through filter paper to procure clear extracts immediately. The filtered biomass was stored at 4 °C for further experiments and used within 10 days.

Biosynthesis of AgNPs: To synthesize nanoparticles, 20 µL $AgNO_3$ solution (50 mM) was added to 980 µL of wheat broth, and then the reactor was exposed to light radiation directly at room temperature. To evaluate the effect of various reaction conditions on AgNP biosynthesis, the samples were set at different light intensity conditions (30,000; 40,000; 50,000; 60,000; and 70,000 lx). The light intensity was detected with a Digital Lux Meter AR823. Control tests were conducted in the dark. Effects of

various wheat straw biomass concentrations (0, 0.5, 1.0, 2.0, and 3.0 mg/mL), NaCl additions (0.5, 1.0, and 1.5 mM), and reaction times (30, 60, 90, and 120 min) were also determined. Particles start sedimenting at the bottom of the beaker after a couple of hours. That indicates the formation of nanoparticles. To separate the nanoparticles from the supernatant, the solution is centrifuged at 15000 rpm for 30min. The supernatant is decanted and the pellet which is a nanoparticle is collected. These formed nanoparticles are stored in an airtight container until further use(39).

12. Biosynthesis of copper oxide nanoparticle from Walnut shell

After collecting the waste walnut shells, they were washed, dried at 90 °C for 6 h in an oven, and pre-treated by crushing mechanically in a mill, followed by sieving to separate 40µm particle size fractions. Ten g of WS and 100 mL of CuSO4 aqueous solution (0.03 M, 0.07 M, and 0.14 M) were mixed at 50 °C for two hours. The reduction of Cu^{2+} was carried out by the drop-wise addition of 0.25 M of $NaBH_4$ (30 mL, 70 mL, 140 mL) to the ethanol mixture. Intensive stirring was continued for three hours. Then the colour of the solution changes to a darker shade. The supernatant and the pellet were formed. The pellet was separated from the supernatant through centrifugation. The pellet is oven-dried for 4hrs at 80°C. Then the copper oxide nanoparticle is stored in a sterilized plastic bottle(40).

13. Preparation of silver nanoparticles fromCoconut shell

Preparation of extract: Coconut shell was collected from a local coconut oil mill and ground into a fine powder by mixer-grinder after hammering. Powdered sample (100 g) was extracted with 500 ml of solvents of increasing polarity (Hexane, Chloroform, Ethyl acetate, and Methanol) in separate flasks and kept overnight at room temperature. Then the samples were filtered and the solvents were dried in a rotary evaporator (under low pressure at 450; 110 C) and re-dissolved in distilled water at 10 mg/ml concentration and used for further analysis.

Synthesis and characterization of AgNPs: The AgNPs was prepared by reduction of $AgNO_3$ using CSE and the conditions were optimized by using 1 ml of $AgNO_3$ (1 mM) mixed with different volumes (100 µl – 900 µl) of extract (10 mg/ml) and made up to 2 ml by using distilled water. The reaction mixer was kept in sunlight (during 12.00 – 1.00 pm) for 1 h and the formation of strong, stable golden brown colour indicated the development of CSE-AgNPs. Synthesized CSE-AgNPshave centrifuged at 10,000 rpm for 10 min and the pellet was re-dispersed in distilled water at 1 mg/ml ratio and used for UV-Spectroscopy, FT-IR, XRD, and TEM analysis(41).

14. Preparation of silver nanoparticles fromCoconut oil cake

Oil cake extraction: Coconut was procured from a local market. The oil cake was suspended in sterile ultrapure water (conductivity = 18 µΩ/m, TOC < 3 ppb) (Barnstead, Waltham, MA), and the flask was shaken at a constant speed of 180 rpm for 2 h. Later, the mixture was filtered through Whatman No. 1 filter paper followed by a 0.2 µm membrane filter. The filtered extract was used for the synthesis of AgNPs.

Synthesis of AgNPs: Briefly, 4 ml of Coconut extract was mixed with 96 ml of 1 mm $AgNO_3$ solution and the resulting milky white mixture was incubated for 8 h in a rotary shaker (180 rpm) at 26 °C. The reduction of Ag^+ ions to Ag nanocrystals was monitored by the change in colour of the reaction mixture from milky white to dark brown. The colour change indicates the formation of the nanoparticles. The newly formed $AgNO_3$ nanoparticles are separated by centrifugation. After centrifugation, the nanoparticles are oven-dried for 6hrs at 100°C. later they are stored in an airtight container(42).

15. Preparation of silver nanoparticles from Banana peel

Banana peel extract (BPE) preparation: Banana (*Musa paradisiaca*) peels were washed and boiled in distilled water for 30 min at 90°C. The peels (100 g) were crushed in 100 ml distilled water and the extract was filtered through a cheesecloth to remove insoluble fractions and macromolecules. This filtrate was treated with an equal volume of chilled acetone and the resultant precipitate was centrifuged at 1000 rpm for 5 min. This precipitate was resuspended in distilled water and stored in a refrigerator at 4 °C for further studies. This extract was used as a reducing as well as a stabilizing agent.

AgNPs Synthesis: 1 ml of BPE (equivalent to 6.8 mg dry weight) is diluted in 50 ml of silver nitrate solution (1 mM). The reaction mixture was incubated in the dark at 30°C to avoid the photoactivation of silver nitrate. After a day the change in the colour of the solution is observed. This indicated the formation of silver oxide nanoparticles. The precipitate is centrifuged to separate from the supernatant. The formed silver oxide nanoparticle is dried in a hot air oven overnight and stored in a container for further studies(43).

XV. Preparation of silver nanoparticles fromTea waste

Preparation of tea extractsynthesis of AgNPs using waste tea extract: Discarded waste tea (10 g) was

immersed in 100 mL of double-distilled water, and the mixture was boiled at 60°C for 5 min. Then the solution was decanted and stored.

synthesis of AgNPs using waste tea extract: For the synthesis of $AgNO_3$ nanoparticles, 20 mL of the waste tea extract was combined with 80 mL of silver nitrate solution (1 $mmol \cdot L^{-1}$) The solution was continued to heat using a water bath, pre-set at 60°C for 30 min and the colour of solution rapidly turns from yellow into brown, showing the realization of AgNPs. The AgNPs were centrifuged for 10 min at 10,000 rpm and the supernatant was decanted off. The precipitation was resuspended with distilled water and again centrifuged three times. The AgNPs thus obtained, were dried in a vacuum oven at 50°Cfor 12h(44).

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