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ORIGINAL RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF STEAM ACTIVATED CHICKEN EGGSHELL FOR GASEOUS POLLUTANT ADSORPTION

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ABSTRACT

Air pollution is a major issue because it can immediately cause health problems. The primary pollutants are oxides of nitrogen, Sulphur, carbon, and oxidants. The collection, preparation, and characterization of chicken eggshell for the production of activated carbon useful in air purification, was carried out. The preparation was done in succession from raw uncarbonized to carbonized and then to steam activated form for adsorption of air pollutants. Powdered Eggshell was Carbonized at different temperatures in the range of 150-600 °C to achieve optimum and effective temperature for best carbon content from its char. The best carbon yield was gotten at 450 °C/1 hour in a carbolite type muffle furnace which gives less room for air flow. Thermal / steam activation of the carbonized eggshell was done at 500 °C for 1 hour. The ash content, bulk density, electrical conductivity, pH, and moisture contents were analyzed respectively. The samples were characterized for their surface area and pore volume. The ash contents for the three samples were in the order Uncarbonized (41.76 %) > Carbonized (32.62 %) > Stean Activated (29.47 %) while the bulk density followed Carbonized (1. 19 g/ml) > Steam Activated (1.11 g/ml) > Uncarbonized (0.79 g/ml). All the three samples had the same value of 0.31 mS/cm for the electrical conductivity while two of the samples (carbonized and activated) had the same pH (8.1) and uncarbonized had a lower pH (7.9). The moisture contents ranged from 2 to 9 % with steam activated sample having the least and uncarbonised, the highest. Tapping density, surface area, pore volume, iodine number, % iodine absorbed values followed the order Steam active > Carbonized > Uncarbonized samples. The steam activated sample having the highest values of all these parameters showed it possesses high adsorption capacity for gaseous pollutants.

1.0 Introduction

Environmental pollution is a worldwide problem and its potential to influence the health of human populations is great. Developmental activities such as construction activities, transportation and manufacturing not only deplete the natural resources but also produce a large volume of wastes that leads to air and water pollution, soil degradation, and oceans; global warming and acid rains. The disposal of untreated waste is a major cause of river pollution and environmental degradation causing ill health and loss of crop productivity (Fereidoun *et al.,* 2007). Additionally, African and Asian countries experiencing rapid industrial growth, and this is making environmental conservation a difficult task (Waziri *et al.,* 2010). Anthropogenic air pollution results from industrial activities, fossil fuel generator fumes, chemical exposures, vehicular emissions and so on.

The pollutant molecules such as Sulphur Oxides (SOx), Nitrogen Oxides (NOx), Lead, Ozone, Benzene, Carbon Oxides (COx), Arsenic, Cd, Hg and Particulate Matters (PM) have a negative effect on human health, which can rapidly spread abroad causing health instability in the system. When the concentrations of these molecules/particles in the air exceed the international standards, the air is considered polluted. Therefore, measures need to be taken to reduce the rate of ambient air pollution if man must survive.

The use of agricultural wastes such as chicken eggshells to remove air pollutants by simple adsorption process cannot be over-emphasized most especially because of the tonne of waste it constitutes to the environment in Nigeria. Hen eggshell typically consists of ceramic materials constituted by a three-layered structure, namely the cuticle on the outer surface, a spongy (calcareous) layer and an inner lamellar (or mammillary) layer (Stadelman, 2000). The spongy and mammillary layers form a matrix composed of protein fibers bonded to calcite (calcium carbonate) crystal. The two layers are also constructed in such a manner that there are numerous circular openings (pores). This structure permits gaseous exchange throughout the shell. The outer surface of the eggshell is covered with a mucin protein that acts as a soluble plug for the pores in the shell. The cuticle is also permeable to gas transmission. The chemical composition (by weight) of by-product eggshells has been reported as follows: calcium carbonate (94 %), magnesium carbonate (1 %), calcium phosphate (1 %) and organic matter (4 %) (Stadelman, 2000). Adsorption is one of the important procedures for the removal of gaseous pollutants from the environment because of its strong affinity and high loading capacity.

Previous studies have proved that Chicken eggshell (ES) is an aviculture by-product that has been listed worldwide as one of the worst environmental problems, especially in those countries where the egg product industry is well developed. In the U.S. alone, about 150,000 tons of this material are disposed of in landfills. ES contains about 95 % calcium carbonate in the form of calcite and 5 % organic materials such as type X collagen, sulfated polysaccharides, and other proteins (Toro *et al.,* 2007; Hussein *et al.,* 2011). The report has shown, among other characteristics that ES has a relatively lower density compared to the mineral calcium carbonate. Eggshell is a biomaterial containing 95 % by weight of calcium carbonate in the form of calcite and 5 % by weight of organic materials, such as (Al_2O_3, SiO_2, S, Cl, P, and Cr_2O_3, MnO) (Hussein *et al.,* 2011). The generalized eggshell structure, which varies widely among species, is a protein linked with mineral crystals, usually of a calcium compound such as calcium carbonate These characteristics qualify ES as a good candidate for bulk quantity, inexpensive, lightweight and low load-bearing composite applications, such as the automotive industry, trucks, homes offices, and factories. Hussein *et al.* 2011 studied the water absorption and mechanical properties of high–density polyethylene/ ES composite, it was found that the addition of eggshell powder to the polymer leads to a decrease in the tensile strength, modulus of elasticity and shore-D hardness. On the other hand, it increases the percentage elongation at break and impact strength. Water absorption of the behaviour of the composites as a function of days was also investigated, and it increases by increasing exposure time for the same filler content.

The adsorption efficiency of eggshell powder for removal of Cu and Pb were studied (Ali *et al.,* 2016). Eggshell powder consists of calcium carbonate, magnesium carbonate, calcium phosphate and protein (Animesh, 2013). The research was a batch scale experiment using different amounts of adsorbent in solution with five different concentrations (5, 10, 20, 40, 100 mg/L) of both metals and mixed combinations. About 92 % to 100 % Cu removal was achieved by using 0.5 to 1.5 g adsorbent for a solution having concentrations of 5 and 10 mg/L of Cu. Two main things were observed from the study which showed that adsorption efficiency depends on the amount of adsorbent as the adsorption efficiency of Pb was increased from 80 % to 100 % in the same solution (5 mg/L), it was also found that adsorption efficiency decreased by 2.5 % and 6.5 % of Cu and Pb to a mixed metal solution. This indicates that the presence of more metals in the solution will decrease the adsorption efficiency. It is therefore clear that eggshell is a good adsorbent for the removal of Cu and Pb and will have a better result by using eggshell in excess (Agarwal, 2013).

The present study is aimed at creating a human-friendly environment by converting common household wastes in Nigeria (chicken eggshell) into useful materials for air purification.

2.0 Materials and Methods

2.1 Materials, Reagents and Equipment

The apparatus and equipment used includes: laboratory glasswares, Laboratory sieve, crucible, desiccator, Stirrer, measuring cylinder, mass balance, pH meter, filtration set-up, Conductivity meter, carbolite muffle furnace, Analytical weighing balance, test tubes, beakers, burette, pipette, conical flask (100 mL and 250 mL), desiccator, electric oven with thermostat, evaporating dish, funnel, graduated cylinder (100 mL), hot plate, magnetic stirrer, mechanical shaker, pH meter, pipette, pipette pump, spatula, standard flasks (1000 mL and 100 mL), suction flask, Whatman filter paper. Reagents used include, potassium hydroxide (KOH), distilled water, sodium hydroxide (NaOH), iodine crystal (I_2), sodium thiosulphate ($Na_2S_2O_7$), Starch, hydrochloric acid (HCl), hydrogen peroxide (H_2O_2), tetraoxosulphate (vi) acid (H_2SO_4), sodium carbonate (Na_2CO_3), potassium iodate (KIO_3), N-(1-Naphthyl)-ethylenediaminedihydrochloride solution, acetone (CH_3COCH_3), Glacial acetic acid (CH_3COOH), sodium di-oxo nitrate (III) ($NaNO_2$), sulphanilic acid ($HNC_6H_4SO_3$), potassium dihydrogen phosphate (KH_2PO_4), disodium hydrogen phosphate (Na_2HPO_4) and methyl orange.

2.2 Sample Collection and Preparation

Chicken eggshells (Figures 1 and 2) used in the experiment were collected free of charge from different hotels, food vendors, kitchens and eateries located within Ilorin, Kwara State, Nigeria. The samples were then washed with distilled water several times to remove dirt particles and dried for three hours in an oven at 150 ^{0}C. it was then allowed to cool to room temperature and subsequently crushed and sieved to prepare uniform size powder. The dried chicken eggshell powder of 2.00 mm particle size was determined using a laboratory tested mesh size sieve. The eggshell powder comprises its shell and membrane, which in turn is made up of 94 % $CaCO_3$, with a small amount of $MgCO_3$, calcium phosphate and other organic matter including protein. Carbonization of powdered eggshells was carried out at a different temperature to obtain an optimum and effective temperature for the best carbon content from its char. Thermal/steam activation of the carbonized eggshell was done at 500 ^{0}C for 1 hour which is the most effective time for steam activation (Schaafsma *et al.,* 2000).

Figure 1: Uncarbonized chicken eggshell

Figure 2: Uncarbonized chicken eggshell granules

2.3 Carbonization and Activation

Carbonization of powdered eggshells (Figures 3 and 4) was carried out at different temperatures to obtain an optimum and effective temperature for the best carbon content from its char. Temperature of 450 ^{0}C for 1 hour in a carbolite type muffle furnace which gives less room for airflow (Rohazriny *et al.,* 2014). Thermal/steam activation of the carbonized eggshell was done at 500 ^{0}C for 1 hour which is the most effective time for steam activation (Tadda *et al.,* 2016).

Figure 3: Carbonized chicken eggshell granules

Figure 4: Steam activated chicken eggshell granules

2.2 Determination of physical properties

2.2.1 Determination of electrical conductivity

Conductance is defined as the reciprocal of the resistance involved and expressed as milli-Siemens per meter or centimeter (APHA, 1995).

$$C = \frac{1}{R} \tag{1}$$

Conductivity was determined by using Hanna Instruments HI-2030-02 Edge Bench Conductivity Meter.

2.2.2 Moisture content determination

The thermal drying method was used to determine the moisture content of the samples. 1.0 g of the (carbonized, uncarbonized and steam activated chicken eggshell) samples were weighed in triplicates. The crucible was placed in an oven at 105 °C to constant weight for 4 hours according to the method of Rengaraj (Ekpete *et al.,* 2011). The difference between the initial and final mass of the carbon represents the moisture content. The percentage moisture content (%) was computed as follows:

$$\text{Moisture } (\%) = \frac{\text{Loss in weight on drying (g)}}{\text{Initial weight (g)}} \text{x } 100 \tag{2}$$

2.2.3 Determination of Bulk Density

A 30 g sample of each powder was weighed out and gently introduced into a 100-ml measuring cylinder. The volume occupied by the powder was noted. This represents the bulk volume of the powder. The cylinder was tapped 200 times on the wooden platform to a constant volume of the powder. The volume occupied by the powder after tapping was noted. This represents the tapped volume. The procedure was repeated three times. Then a weighted (known) sample was placed in a dry measuring cylinder and tapped until the carbon samples occupy a minimum volume known as the Bulk volume (Bv), this was read from the calibration.

The bulk density (BD) was then calculated from the equation. (g/ml).

$$BD = \frac{M_2 - M_1}{B_V} \text{ or } \frac{\text{mass}}{\text{volume}} \tag{3}$$

M_1= mass of measuring cylinder in grams
M_2= mass of measuring cylinder + its contents
V= volume of the measuring cylinder

2.2.4 Tapping Density (T_D)

The tapped density (g/ml) is an increased bulk density attained after mechanically tapping a container containing the powder sample. The tapped density is obtained by mechanically tapping a graduated measuring cylinder or vessel containing the powder sample.

$$T_D = \frac{\text{mass (g)}}{\text{Tapped volume (ml)}} \tag{4}$$

2.2.5 BET surface area (BET)

Brunauer, Emmett and Teller are the three men who proposed a theory to measure the surface area of porous powder-type solid particles. The principle involved is the adsorption of gas molecules to the surface of the solid whose surface area is required. From the area of each molecule, the whole area of the solid can be calculated. BET theory is based on multilayer adsorption with considering the following assumptions:

a. Gas molecules can be physically adsorbed on the solid surface and form infinite layers
b. There is no interaction between layers
c. Langmuir theory is applied to each layer

2.3 Determination of Chemical Properties

2.3.1 Determination of pH

The pH meter was first calibrated, and then the standard test method for the determination of activated carbon pH ASTMD3838-80 was used. 1.0 g each of uncarbonized, carbonized and activated carbon eggshell was weighed and transferred into a beaker. 100 ml of distilled water was measured and added and stirred for one hour. The samples were allowed to stabilize before the pH was measured using a pH meter Hanna instruments, portable pH meter, H1981-6 PH/EC/TDS Temperature (Ekpete *et al.,* 2011).

2.3.2 Ash content determination

For ash content determination, 1.0 g of each sample was transferred into the crucibles. The crucibles containing the samples were placed in the furnace and the temperature was increased and samples heated to 500 °C for 1 hour 30 minutes, then removed and allowed to cool in a desiccator to ambient temperature and weighed. The ash content was calculated using the equation below (Ekpete *et al.,* 2011).

$$\%\,\text{Ash} = \frac{\text{Ash weight}}{\text{Oven dry weight}} \times 100 \tag{5}$$

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2.2.3 Volatile matter determination

Volatile matter was calculated using the equation (Ekpete *et al.*,2011).

$$\frac{\text{Weight of volatile component}}{\text{Oven dry weight}} \times 100 \tag{6}$$

2.3.4 Determination of iodine number

0.5 g AC from each sample from the determination of volatile matter was weighed into a beaker and 25 ml of standard Iodine solution (0.0229 M) confirmed concentration after standardization was added. The mixture was swirled vigorously for 10 minutes and filtered using a funnel impregnated with clean filter paper. 20 ml of the clear filtrates was titrated with the standard (0.1115 M) thiosulphate confirmed concentration after standardization to a persistence of a pale-yellow color. 5 ml of freshly prepared Starch indicator solution was added, and titration resumes slowly until a colorless solution appeared, the procedure was repeated for the remaining samples. The titration was also repeated with 20 cm^3 portions of the standard iodine solution (not treated with AC from the precursor) to serve as blank titration. The iodine adsorption number (IAN) was calculated from the relationship as equation below (Itodo *et al.*, 2010).

$$\mathrm{IAN} = \frac{M_S(V_b - V_s)}{2M_a} \tag{7}$$

Where: M_s = molarity of thiosulphate solution (mol/dm^3).
V_s = volume of thiosulphate (cm^3) used for titration of the PAC aliquot.
V_b = volume of thiosulphate (cm^3) used for blank titration.
M_a = mass of AC (g)

2.3.5 The Percent Iodine Adsorbed

The percent iodine adsorbed by each carbon was calculated by applying the formula below:

$$\frac{\text{mL of } Na_2S_2O_3 \text{ in blank} - \text{mL of } Na_2S_2O_3 \text{ in sample}}{\text{mL of } Na_2S_2O_3 \text{ in blank}} \times 100 \tag{8}$$

Iodine number is usually used to roughly estimate the surface area of activated carbon at room temperature conditions. It is used as an indicator for the porosity and adsorbent capacity of the activated carbon. The higher iodine number of carbons has been attributed to the presence of large micropore structure and the great probability of carbons having a large surface area due to enlargement of their pore structure (Ekpete *et al.*, 2011).

2.3.6 Fixed carbon content

$$\text{Fixed carbon FC} = 100 - (\%\text{ moisture content} - \%\text{ volatile matter} - \%\text{ ash content}) \tag{9}$$

2.3.7 Scanning electron microscopy (SEM)

SEM is one of the most versatile instruments available for the examination and analysis of microstructure morphology. This technique was used to study the morphological feature and surface characteristics of the selected materials (Kumar, 2021).

3.0 Results and Discussion

3.1 Electrical Conductivity and pH

The pH used was of buffer 7 solution. The pH of the solution is a major factor in determining adsorption. pH can influence surface electric charge of the sorbent. The pH dependent variation of the potential between liquid and solid phases may be due to the polarities and deprotonation of functional groups on the sorbent surface. Generally, acidic species adsorbed better at low pH, while basic species adsorb better at higher pH. Table 1 showed the pH and conductivity values of the three samples.

Table 1: Values for pH and electrical conductivity

Eggshell	Uncarbonized	Carbonized	Steam activated
pH	7.9	8.1	8.1
Conductivity ($mScm^{-1}$)	0.31	0.31	0.31

The conductivity values are similar for all three powdered samples. The pH values of all three samples are greater than 7 which showed that they are basic in nature. According to Ahmedna, (2000) as pH increases adsorption increases, therefore carbonized and steam activated eggshells will have the highest adsorption efficiencies respectively compared to uncarbonized eggshells.

3.2 Physical and Chemical Properties

The results of physical and chemical properties of uncarbonized, carbonized and steam activated eggshells are shown in Table 2.

Properties/(Unit)	Uncarbonized chicken eggshell	Carbonized chicken eggshell	Steam activated chicken eggshell
Moisture content (%)	9	2	5
Bulk density (g/ml)	0.79	1.19	1.11
Tapping density (g/ml)	1.23	1.20	1.25
Volatile content (%)	17.98	_____	_____
Ash content (%)	41.76	32.65	29.47
Iodine number (cm^3/g)	0.207	0.276	0.322
Iodine adsorbed (%)	22.2	29.6	34.6
Fixed carbon content (%)	31.26	_____	______

Table 2: Results of physical and chemical properties of the three samples

From Table 2, the moisture content of steam activated eggshell showed considerable amount of moisture which makes it fit for adsorption. Ash content of steam activated eggshell showed the least, which makes it desirable for adsorption, because the lesser the ash content, the higher its adsorption potential. Volatile matter and Fixed Carbon were gotten only for Uncarbonized eggshells.

Bulk density tests for the flow consistency and packaging quantity of solid samples. Generally, well aggregated, porous materials have lower bulk density. However, bulk density is important because the higher the bulk density is, the better the filterability and the better the adsorbing properties of the activated carbon (Erhayem, 2015). This implies that both carbonized and steam activated eggshell will be suitable for adsorption.

Tapped density is an increased bulk density attained after mechanically tapping a container containing the powder sample. The tapped density is obtained by mechanically tapping a graduated measuring cylinder or vessel containing the powder sample. Because the interparticle interactions influencing the bulking properties of a powder are also the interactions that interfere with powder flow, a comparison of the bulk and tapped densities can give a measure of the relative importance of these interactions in a given powder. Such a comparison is often used as an index of the ability of the powder to flow. Uneven powder flow can result in excess entrapped air which can affect the amount and quantity of adsorbent that will contact contaminants which in turn gives rise to poor adsorption rate or capacity. Steam activated chicken eggshell has the highest tapped density, hence it is most suitable for adsorption.

From the results showed in table 2, the iodine number calculated showed that steam activated eggshell has the highest number at 0.322 cm^3/g. This showed that there is an increased number of micro-pore spaces after the steam activation process carried out on the sample. For percent iodine adsorbed, steam activated chicken eggshell showed the highest percentage of iodine adsorbed, which implies that it's the best material for adsorption out the 3 samples.

3.3 SEM Analysis

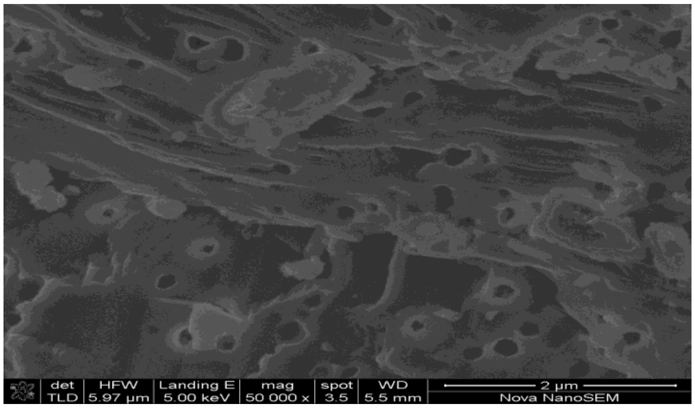

Figure 5: SEM image of Uncarbonized

Figure 5 showed the SEM micrograph of uncarbonized eggshell of a magnification of 50, 000 on 2 micron with the surface showing several irregular structures with layers of porous structure spread all over the surface. These pores are majorly macro in nature with little micro-pores. This in turn is responsible for the poor iodine adsorption rate, for iodine adsorption is on to micro-pore and not macro-pores.
Adsorption of gaseous pollutants are also to micro-porous structures which implies that uncarbonized eggshell is a poor adsorbent for the adsorption of gaseous pollutants.

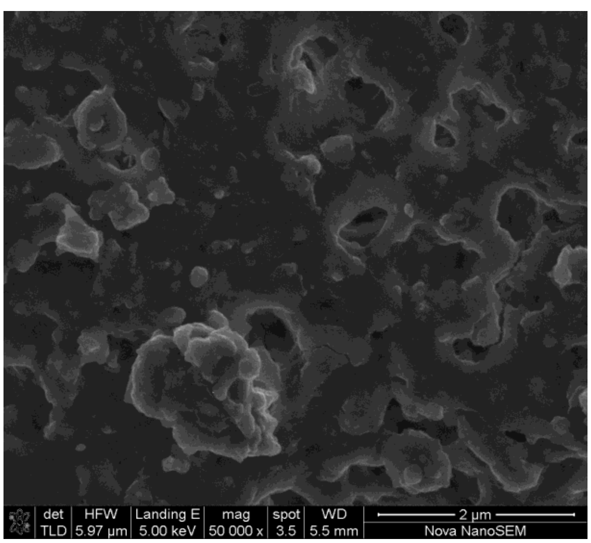

Figure 6: SEM image of carbonized

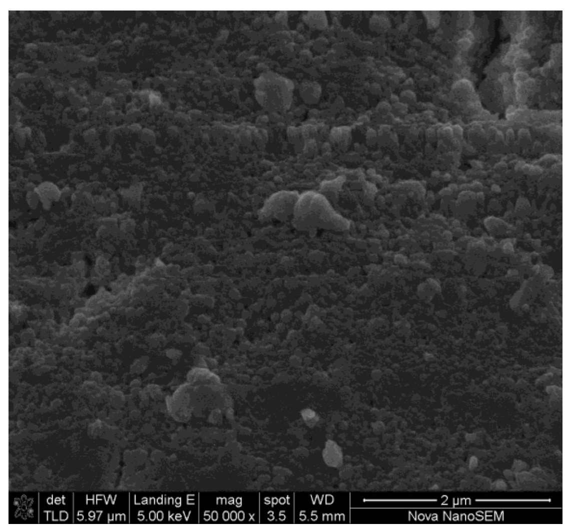

Figure 7: SEM image of Steam activated

Figure 6 showed the SEM micrograph of carbonized eggshell of a magnification of 50,000 on 2 micron with the surface showing several irregular structures with layers of porous structures. Here, there is a change in surface morphology as a result of carbonization of the eggshell granules. This displays a mesoporous structure which is a very poor adsorption surface for gaseous pollutants, although it is also a viable adsorption surface for other forms of materials but not gaseous pollutants.

Figure 7 showed the SEM micrograph of steam activated eggshell at a magnification of 50, 000 on 2 micron with the surface showing a considerable change in the surface morphology as a result of steam activation after it has been carbonized. It has improved micro-porous structure with fine pores and cracks. These pores provide good possibility for adsorption of gaseous pollutants, and this makes steam activation of the eggshell of an advantage and a success since it was able to give an improved sample adsorption purpose.

3.4 BET Analysis

BET surface area and pore volume of uncarbonized, carbonized and steam activated chicken eggshells are shown in table 3.

Table 3: BET Result of the three samples

Properties/(Unit)	Uncarbonized chicken eggshell	Carbonized chicken eggshell	Steam activated chicken eggshell
Surface area (BJH) (m^2/g)	3.242	5.760	6.835
Pore volume (cc/g)	1.580	2.818	3.221
Pore size (nm)	2.115	2.421	1.793

From the above BET result of BJH analysis (Table 3). It showed that steam activated eggshell has the highest surface area and pore volume, this is responsible for the differences in adsorption capacity. Hence, steam activation is integral aspect of pore improvement for adsorption of gaseous pollutants thereby making steam activated chicken eggshell the best for adsorption of gaseous pollutants.

4.0 Conclusion

From the preparation, characterization, physicochemical analysis, BET and SEM analysis carried out on uncarbonized, carbonized and steam activated chicken eggshells which is a major waste material in our environment; It was observed that steam activation of chicken eggshell favours and improves the pore spaces, pore volume, surface area, overall morphology, and adsorption capacity of chicken eggshell powder. According to ISO 15901-3:2007(en) standards for micropores, mesopores and macropores, steam activated chicken eggshell showed largest amount of micropores sizes (surface area and pore volumes), thereby making it highly desirable for adsorption of gaseous pollutants since 1.00 nm is the minimum pore diameter for adsorption of SO_X and NO_X.

Hence, steam activated chicken eggshell is better than all the other three processed types of chicken eggshell powder for adsorption of gaseous pollutants.

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