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RAPID METHOD FOR DETERMINATION OF NANO SURFACE AREA OF ARECANUT SHELL DERIVED ACTIVATED CARBON BY IODINE ADSORPTION NUMBER

Abstract. Activated carbon is the most versatile and commonly used adsorbent. Activated carbon is prepared for 5e+10ng , 1e+11ng, and 3e+11ng batch size. Impregnation ratio maintained is 1:1,2:1,3:1,4:1.Nano activated carbon from arecanut shell is derived. Particles size diameter maintained to 53000 nm. In this study the specific surface area determination of activated carbon by means of the low-temperature argon adsorption (the BET method) is compared with the measurement of the surface area based on the adsorption of I_2 from the aqueous KI solution. The iodine adsorption number for the BET surface area is calculated. It is predicted iodine adsorption number S_{IN} method can be used for a quick estimation of the structure development of porous carbonaceous materials.

Keywords: Activated carbons, Iodine adsorption number (S_{IN}), Specific surface area (S_{BET}).

1.Introduction

First of all, raw material of the activated carbon is acquired by collecting arecanut shell store from Bangalore and Kerala.Highly porous carbon can be produced from a variety of natural and synthetic precursors [1,2] In its original state, the surface of a carbon is energetically heterogeneous [3], but as discovered by Beebe et al. [4] the heterogeneity is considerably reduced by heat treatment in an inert atmosphere. Precursor used for the production of activated carbon in this study is arecanut shell Activated carbon produced from residues would reduce the pressure on forests since wood is also commonly used for this purpose [5]. Many agricultural by-products such as coconut shell [6,7], grain sorghum [8], coffee bean husks [9], rubber wood sawdust [10], chestnut wood [11], have been discovered to be suitable precursors for activated carbon due to their high carbon and low ash contents

2. Experimental

2.1 Preparation of activated carbon

The carbonization Arecanut shell biomass is performed under a nitrogen flow of 100 cm^3 min−1 STP for 2hr. After activation, the activated carbon product removed and subsequently cleaned by removing the fibers and washing several times with distilled water to remove impurities The arecanut shell is chopped to pieces of ¼ inches ,then dried at 110 °C until constant weight of the sample is reached.. Then, dried and size-reduced arecanut shell is kept in a muffle furnace as raw material for activated carbon production.

Chemical activation method using phosphoric acid is used to activate the raw material. 5e+10ng, 1e+11ng,and 3e+11ng of raw material is impregnated by certain amount of 85 wt.% concentration phosphoric acid with occasional stirring. The amount of phosphoric acid solution used is adjusted to give a certain impregnation ratio (weight of activating agent/weight of raw material) of 1:1, 2:1, 3:1, and 4:1. The resulting slurry is then kept in a desiccator Overnight .

After 24 h, the mixture of raw material and phosphoric acid is then ready to have two-stage activation process with semi-carbonization as first stage [8]. In the first stage, the slurry is put in a horizontal tubular reactor and kept in a muffle furnace to experience semi-carbonization at a temperature 200 ∘C for 30 min. After semi-carbonization, the black and sticky dry powder is heated until certain activation and cooled in a desiccator. The activated carbon product is then repeatedly washed with warm distilled water (70 ∘C) until constant pH of the solution is reached. Finally, the activated carbon is dried in a vacuum oven at 110 ∘C for 24 h. The activated carbon is then stored in a desiccator for later experiment use. Finally the particle size maintained at 53000 nm.
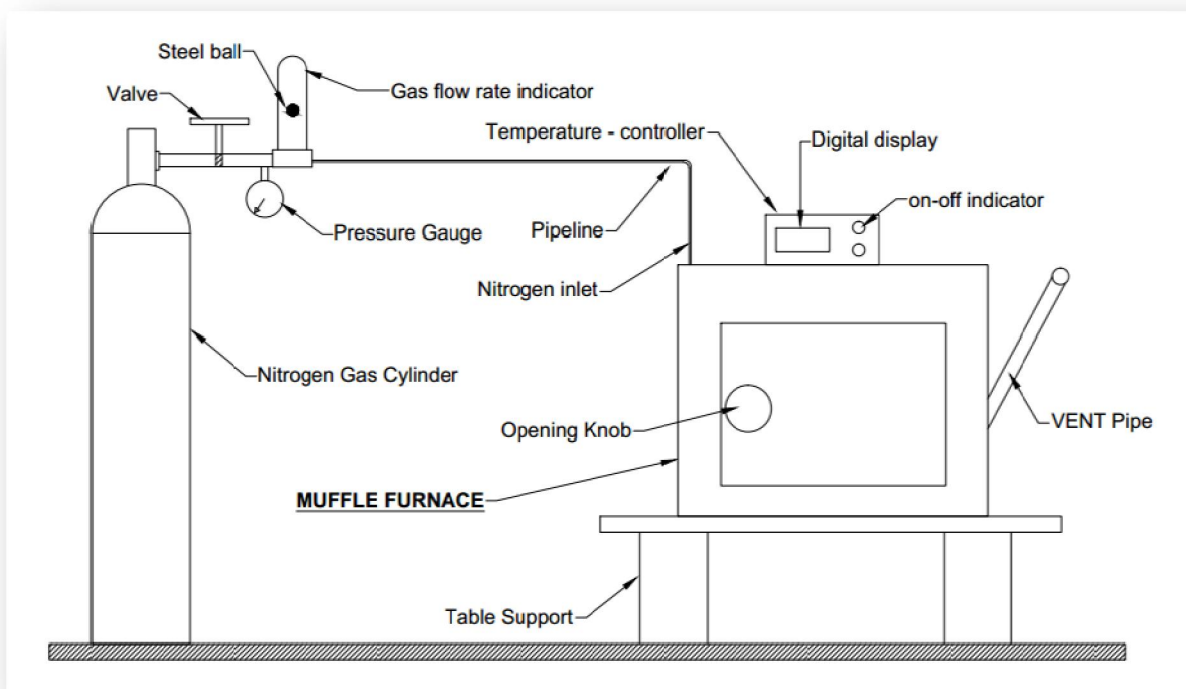
Experimental is as shown in fig .1

Fig: 1 Experimental Set Up

4.Result and Discussion

4.1 Yield of activated carbon

In activated carbon preparation, yield is usually defined as final weight of activated carbon produced after activation, ishing, and drying, divided by initial weight of raw material; both on a dry basis [6]. Table.1 shows the yield of activated carbon.It is observed , yield of AC is increased with increase of impregnation upto 3:1 ratio and decreases from 3:1-4:1 impregnation ratio for each batch i.e 5e+10ng, 1e+11ng,and 3e+11ng

Table 1 - Yield of activated carbon prepared from biomass at 400^0C from Arecanut Shell

Sample	Yield% (5e+10ng)	Yield% (1e+11ng)	Yield% (3e+11ng)
AS-PA-01 (1:1)	60.6	65.3	50.3
AS-PA-02 (1:2)	77.2	77	73.33
AS-PA-03 (1:3)	84	88	83.33
AS-PA-04 (1:4)	70	80	75.23

This is due to binding of O and H atoms decreases from 3:1 impregnation ratio. This led to decrease yield.The carbonization yield depends on the amount of carbon removed by binding with O and H atoms (Caturla et al., 1991).

4.2 Determination of Iodine Number

This is the most fundamental parameter used to characterize activated carbon performance. It is a measure of activity level (Higher degree indicates higher activation), often reported in mg/g (with typical range of 5e+8 – 1.2e+9 ng/g). It is a measure of the microspore content of the activated carbon (values > 0 to 20 AO, or up to 2nm) by adsorption of iodine from solution. It is equivalent to surface area of activated carbon between 9000 nm^2 /g and 11000 nm^2 /g and. (Elliot et al., 1989). It tells of carbon that preferentially adsorb small molecules. High value indicate high degree of activation (Aziza et al.,2008;Elliot et al.,1989).

Table no: 2 Iodine number value of Arecanut shell

Sample	Iodine Number (5e+10ng))	Iodine Number (1e+11ng)	Iodine Number (3e+11ng)
AS-PA-01 (1;1)	692.81	829.63	812
AS-PA- 02 (1:2)	706.67	937.93	848
AS-PA- 03 (1:3)	822.57	976.6	906
AS-PA- 03 (1:4)	800.10	900	880

4.3 Determination of Iodine number surface Area

The aim of the current work is to calculate the surface area per iodine atom (ω_I), and then to determine the correlation between the specific surface area measured by the method of the low temperature argon adsorption (S_{BET}) and the surface area (S_{IN}) measured by the iodine adsorption number (IN) for activated carbons.

The analysis of the experimental adsorption isotherms (BET) in the relative pressure range of p/po = 0.05–0.4 allows determining sorption capacities (a_m) which can then be used to calculate the specific surface area according Eq. (1)

$$S_{BET} = a_m \cdot N \cdot \omega_{Ar} \tag{1}$$

In this work, we assume the ω_{Ar} value equalled to equivalent surface measured according to the nitrogen standard. a_m: monolayer capacity, mole/g, N: Avogadro constant, N = 6.023 × 10^{23} 1/mole ω_{Ar} : surface area occupied by one of adsorbate (argon) atom

The determination of the iodine number is one of the methods often adopted in the industry utilizing activated carbons If we take into account the definition of the iodine adsorption number by the analogy to Eq. (1), Thus:

$$S_{BET} = \frac{IN \cdot 10^{-3}}{M_I} \cdot N \cdot \omega_I + \Delta S \tag{2}$$

Table no: 3 Iodine adsorption number of Arecanut shell

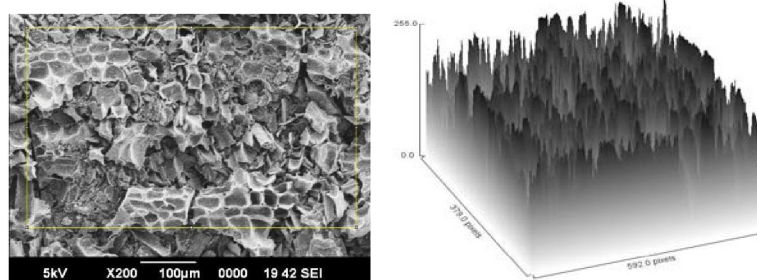| Sample	S_{IN} (5e+10ng)	S_{IN} (1e+11ng)	S_{IN} (3e+11ng)
AS-PA-01(1;1)	496.06	653.45	639.56
AS-PA- 02(2:1)	556.57	738.71	667.88
AS-PA- 03(3:1)	647.85	769.46	713.56
AS-PA- 04(4:1)	630.15	708.84	693.08

Fig: 2 - SEM of Arecanut shell carbon Fig: 3 Particle distribution image of ASAC

From table 3 iodine adsorption number shows same effect as shown by iodine number Value of iodine adsorption number increases uptill 3:1 and decreases from 3:1-4:1 impregnation ratio .Standard determination of the iodine adsorption number comprises the measurement of iodine amount in the adsorption layer of an activated carbon sample (in mg iodine/g adsorbent). The change of the bulk concentration results in the changes in the composition of the interfacial layers, which induces mutual displacements of the solution components from the adsorbed layer (Jankowska et al., 1991).

During excess adsorption from the solution there are no unoccupied sites on the surface, which implies that the same iodine amount occupies the same surface in the different carbon samples. It is worth noting that the specific surface area has been used most often for the characterization of the different porous solid bodies.

5.Conclusion

Arecanut shell derived activated carbon prepared in the form of the fine-grained ($<$0.080 mm) carbon samples, the iodine adsorption number in a range of IN = 496060000–769460000 ng/g can be recalculated into the specific surface area according to Eq. (2). If we analyze the procedure for the determination of the iodine adsorption number according to (PN-83/C–97555.04), this number equals the specific surface area S_{BET} . The determined iodine surface area, which amounts to ω_I = 0.2096 nm^2, is a rough evaluation and has been calculated in proviso that iodine is covered hexagonally on the adsorbent surface .Iodine adsorption number to the specific surface area can be a rapid and efficient method for the evaluation of the surface area with a systematic error of a few nm^2/g in relation to the S_{BET} value.

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