



Characterization of Activated carbons Prepared from a Locally Available Material by Iodine Number

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Abstract: The adsorption of Iodine by the activated carbons prepared from a locally available material Lapsi Seed Stone has been studied. Various activated carbons were prepared by varying the parameters such as ratio of Lapsi Seed Stone particles to Phosphoric acid, percentage of phosphoric acid concentration, carbonization temperature and carbonization time. Determination of Iodine numbers of the activated carbons was done by the standard method. The effect of various preparation conditions on Iodine number of different activated carbons is also investigated. The optimum conditions for the preparation of activated carbon are found to be 400 °C -the carbonization temperature, 4 hours - the carbonization time, 1:1- the ratio of Lapsi Seed Stone particles and Phosphoric acid and 50 %-the percentage of phosphoric acid concentration

Keywords: Lapsi seed stone, activated carbon, adsorption, iodine number

1. Introduction

Activated carbon is carbonaceous material that has well developed pores, internal surface area and relatively high mechanical strength. It has been widely used in waste water and gas treatments as well as in catalysis. Activated carbon has many important uses including solution purification, removal of tastes and odor from domestic and industrial water supplies, vegetable and animal fats, oils, alcoholic beverages, chemicals, pharmaceuticals, removal of heavy metals and dye from water etc [11]. Most of activated carbons are prepared by a two- stage process carbonization followed by activation. Carbonization is done to enrich the carbon content and to create an initial porosity and activation process helps in enhancing the pore structure. The activation can be carried by two different processes physical and chemical. In physical activation carbonization and activation are carried out separately. The carbonization so called pyrolysis is carried out at high temperature in inert atmosphere in order to eliminate maximum of oxygen and hydrogen elements. The second one is thermal activation at the same temperature for pyrolysis or at a higher temperature in the presence of oxidizing gases steam or carbon dioxide or both. By chemical activation it is possible to prepare activated carbon in only one step. Pyrolysis and activation are carried out simultaneously in the presence of dehydrating agents (e.g. $ZnCl_2$, H_3PO_4 , H_2SO_4 , KCl etc.). In this study preparation of activated carbons from Lapsi Seed Stone by chemical activation with phosphoric acid was carried out.

A large number of agricultural wastes have been used for the preparation of activated carbon. The waste materials such as apricot stones [6], date stones [2], guava seeds [3] black stone cherries [7], peach stones [1 & 4], orange peel [5], Peanut shell [13] are the some examples of low-cost accessible waste materials for the production of activated carbons. Lapsi seed stone is the waste product of Lapsi fruits. Lapsi (*Choerospondias axillaris*) belongs to the family Anacardiaceous. It is a large, fruit bearing deciduous tree native to the hills of Nepal (865-1900m). Lapsi fruit is consumed fresh, pickled or processed into a variety of sweet and sour fruit products locally called "Mada" or "Titaura". It is a rich source of vitamin C. It is also believed to aid in digestion and is often consumed after a protein rich meal. Seed stones are used as fuel in brick kilns in the factories and the trunk of the tree is used as fuel wood and timber [8&9]. Iron impregnated activated carbon prepared from Lapsi seed stone had been investigated for the removal of arsenic from water [10].

Not much information is available regarding the preparation of activated carbon from Lapsi Seed stone using phosphoric acid as an activating agent. Activated carbon was prepared from Lapsi seed stone by chemical activation with zinc chloride for the adsorption of arsenic from water. No report is available in the literature about the preparation of activated carbon from Lapsi seed stone using phosphoric acid as an activating agent. This lack in existing literature is a motivation for the present study.

Activated carbons are usually characterized by several physical parameters such as surface area and pore volume. The surface area according to BET is then determined. The surface areas of activated carbons are usually measured using the Brunauer-Emmett-Teller (BET) method, which employs the nitrogen adsorption at different pressures at the temperature of liquid nitrogen (77 K). The surface area according to BET is then determined by the product of the cross-sectional area of the nitrogen molecule, of the Avogadro's number and of the specific monolayer capacity of nitrogen, which is obtained by an equation proposed by BET with further modifications. For the pore volume determination, the procedure more commonly used also uses nitrogen adsorption isotherm data. The total pore volume is estimated from the amount of nitrogen adsorbed at the highest relative pressure and the micropore volume is calculated from the nitrogen adsorption isotherms using the Dubinin-Radushkevich equation. In spite of these methods being more used and employed as a reference in the surface area and pore volume determination of porous materials, they are time consuming and require the use of expensive equipment.

Additional information about the structure of activated carbons can be obtained by the adsorption characteristics of different adsorbates, such as methylene blue and iodine. Iodine being small molecules enters into micropores while methylene blue being a big molecule is absorbed into the mesopores. Many carbons preferentially adsorb small molecules. Iodine number is the most fundamental parameter used to characterize activated carbon performance and is defined as the number of milligrams of iodine adsorbed by one gram of carbon [12]. It is a measure of activity level (higher number indicates higher degree of activation), often reported in mg/g (typical range 500-1200 mg/g). It is a measure of the micropore content of the activated carbon (0 to 20 Å, or up to 2 nm) by adsorption of iodine from solution. The aim of this investigation was to determine Iodine number of activated carbons prepared from Lapsi Seed Stone by chemical activation with Phosphoric acid

2. Material and Methods

2.1 Preparation of Adsorbent

The precursors used in the preparation of activated carbon are seed stones of Lapsi fruits. The fruits were collected from Fruits and Vegetable Market, Kalimati, Kathmandu. The fruits are boiled for the pulp and then seed stones are exposed and collected. The seed stones were washed well with tap water and distilled water, dried at 110 °C and crushed to desired size (300µm) using a grinder crusher. The crushed particles were then sieved to obtain the fraction 300 µm. Various activated carbons were prepared by varying the parameters such as ratio of Lapsi Seed Stone particles to Phosphoric acid, percentage of phosphoric acid concentration, carbonization temperature and carbonization time. The activated carbons prepared were cooled at room temperature and were washed several times with distilled water. The materials were then dried at 110 °C for 24 hours, cooled and sieved to obtain the particles of size 106 µm.

2.2 Chemicals

All chemicals and reagents were of analytical grade (Merck and Qualigens Company). All the working solutions were prepared by diluting the stock solutions with distilled water.

2.3 Iodine number

The adsorption of iodine is considered a simple and quick test for evaluating the area of activated carbons associated with pores larger than 1 nm. The iodine number, defined as the amount of iodine adsorbed per gram of activated carbon at an equilibrium concentration was measured according to the procedure established by the American Society for Testing and Materials (ASTM 2006). 100 mg of activated carbon was added to 5ml of 5% HCl and boiled. The solution was cooled at room temperature and added 10 ml of 0.05 M iodine solution. The solution was shaken for 1 min, filtered and washed with distilled water. The whole of the filtrate was titrated against 0.05 M sodium thiosulphate solution using starch as an indicator. The iodine number was calculated by the following formula.

Iodine number = C × Conversion factor

The conversion factor can be calculated as follows:

C = Blank reading - Volume of Hypo solution consumed after the adsorption by Activated carbon

3. Results and Discussion

Activated carbons were prepared from Lapsi (*Choerospondias axillaris*) seed stone by chemical activation with Phosphoric acid. The resultant carbons were characterized by Iodine number. Effect of various preparation parameters like ratio of Lapsi Seed Stone to Phosphoric acid, carbonization time, carbonization temperature and percentage of phosphoric acid concentration were studied.

3.1 Effect of Carbonization temperature and carbonization time

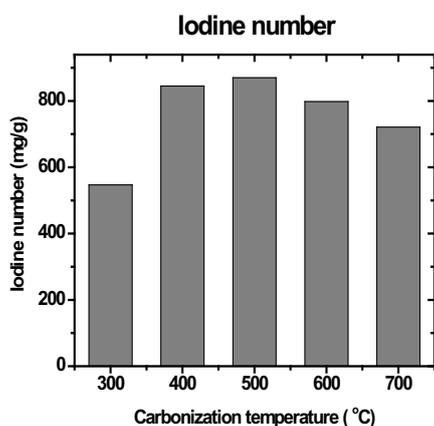


Fig. 3.1: Effect of carbonization temperature on Iodine number of activated carbons

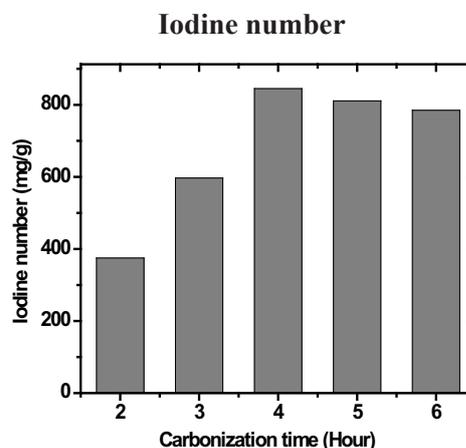


Fig. 3.2: Effect of carbonization time on Iodine number of activated carbons

The variation in iodine number of activated carbon as a function of carbonization temperature keeping other parameters such as impregnation ratio, carbonization time and concentration of phosphoric acid constant is shown in Fig. 3.1. From the Figure it is clear that iodine number increases up to 500 °C and then decreases. Iodine is a small molecule with minimum pore diameter 10 Å. Iodine number gives approximate measure of the micropore content.

The results indicate that the carbonization temperature has a significant effect in the pore structure of activated carbon which ultimately determines the adsorption capacity of activated carbon. The increase in iodine number with increase in carbonization temperature may be due to the creation of more micropores and development of unsaturation in the activated carbon.

The effect of carbonization time on Iodine number of the activated carbons keeping other parameters like impregnation ratio, carbonization temperature and concentration of phosphoric acid constant is shown in Fig. 3.2. The Iodine number increases with carbonization time up to 4 hours and then after iodine number decreases

The increase in iodine number up to 4 hours may be due to escaping of tar from raw materials. After 4 hours no further development of micropores takes place but there may be widening of micropores resulting in the formation of mesopores. From this result one may conclude that 4 hours of carbonization at 400 °C is necessary to prepare the activated carbon with high iodine number.

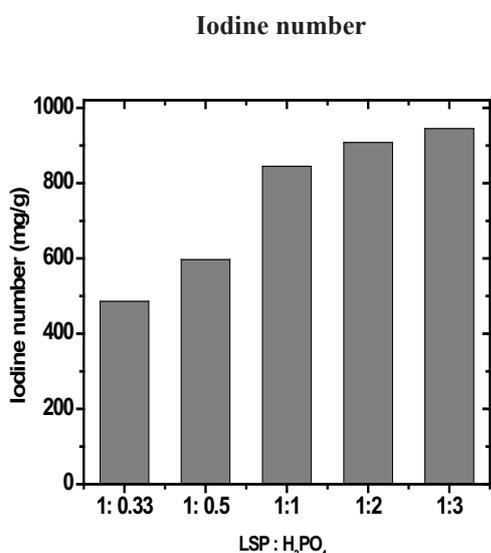


Fig. 3.3: Effect of LSP: H₃PO₄ on Iodine number of activated carbons

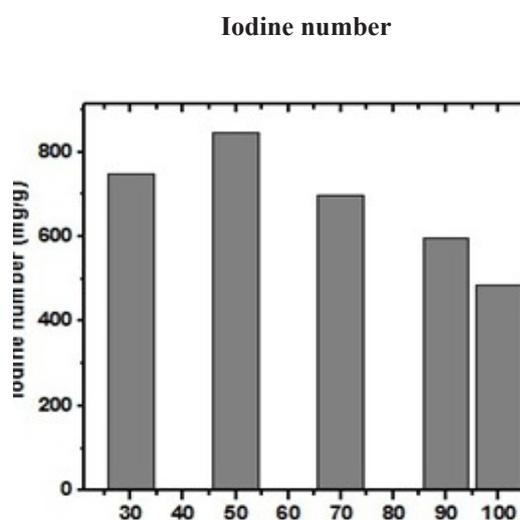


Fig. 3.4: Effect of concentration of H₃PO₄ on Iodine number of activated carbons

3.2 Effect of Ratio of Lapsi Seed Stone Powder: H₃PO₄ and Concentration of Phosphoric acid

The variation of Iodine number of the activated carbons prepared at different LSP: H₃PO₄ ratio keeping constant other parameters is shown in Fig. 3.3. The iodine number of activated carbons increases with the increase in amount of acid and after 1:1 ratio the increase is not significant. This may be due to the degeneration of pores caused by excessive dehydration with more acid

Iodine number of the activated carbons prepared by activation with different concentration of H₃PO₄ keeping other parameters constant is shown in Fig. 3.4. When the concentration of H₃PO₄ increases from 30 % to 50 % H₃PO₄ iodine number increased significantly and then after from it decreases gradually. The decrease in iodine number at high concentration of phosphoric acid may be caused by degeneration of porous structures of activated carbons due to excessive dehydration.

4. Conclusion

The determination of Iodine number is the simple and quick method to characterize activated carbon among various activated carbons prepared. Procedure used in the determination of Iodine number is relatively cheap and simple and do not require the use of sophisticated equipment. Although this method does not substitute the proper characterization of the textural properties for activated carbons, it is valuable for laboratories in which the equipment for gas adsorption experiments is not available. The optimum conditions for the preparation of activated carbon from Lapsi Seed Stone are 400 °C -the carbonization temperature, 4 Hours - the carbonization time, 1:1 – the ratio of Lapsi Seed Stone particles and Phosphoric acid and 50 % - the percentage of phosphoric acid concentration.

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