Collapse Strength of Thermal Black Briquettes Containing Varying Amounts of Adsorbate

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Abstract. The objective of these experiments was to determine the validity or non-validity of using adsorption data obtained on spherical, non-porous carbon powders to intrepret the adsorption process on porous activated carbons. The experimental procedure involved compressing Huber N326 (26 mm) thermal black into cylindrical briquettes, and after adsorption of varied amounts of carbon tetrachloride or water, determining the collapse strength. The results supported the contention that the transfer of data was invalid.

Introduction

Adsorption researchers have been using data obtained on non-porous, spherical grained carbon blacks to interpret the adsorption process in porous activated carbons. The first assumption is that the carbon black particles are sufficiently separated so that multilayer adsorption (BET theory) can proceed without interaction between particles. The second assumption is that whatever happens on a carbon black surface also happens in the same manner on the surface of a porous activated carbon. A different point of view, held by the author of this paper, is that the carbon black particles are close enough for capillary condensation to occur just as it does in activated carbon, and that the isotherm characteristics are determined by the pore structure. The objective of this study is to generate evidence that the latter is the more correct viewpoint.

Carbon Properties

The carbon used was Huber N326 thermal black which was reported to consist of non-porous, spherical particles, 260Å average diameter. The specific gravity was 1.8 g/cc and surface area by nitrogen was 80 m^2/g. To prepare thermal black for testing, it was leached in succession with carbon tetrachloride, acetone, and water, and then ground under water in a motor-driven mortar and pestle. The ground material was classified into size fractions by settling in water, giving a range of aggregate sizes of roughly 10,000 to 52,000Å. Aggregates smaller than 10,000 Å recombined to form larger ones.

Briquetting Procedure

Figure 1 shows the apparatus for briquetting and collapse strength measuring. A weighed amount of dry thermal black is compressed in the glass tube at 2065 g/cm^2 by means of the glass plunger and weight. The briquette is then forced out of the glass tube with the plunger and retained in the weighing bottle, wherein it may then be strength tested when dry or treated with carbon tetrachloride or water before strength testing. The briquettes were always strength tested in the dry state between each test with adsorbate to make certain the thermal black properties were not changing.

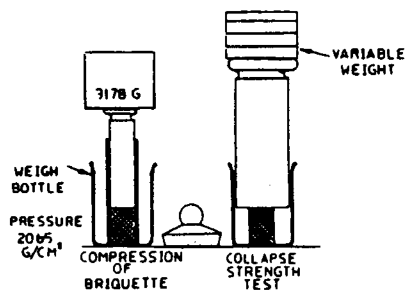

Figure 1. Apparatus for briquette formation and collapse strength test.

Properties of the Briquettes

The weight of carbon used was kept near 1.68 g which gave a briquette 1.85 cm in length when applied pressure was 2065 g/ cm^2. The diameter was 1.4 cm and volume, 2.85 cc. Per cc, the carbon weight was 0.59 g and carbon volume, 0.33 cc. This gives a void volume of 0.67 cc/cc which is considerably larger than 0.25 cc/cc, the void volume when closest hexagonally packed. The presence of the aggregates greatly increases the void volume.

Figure 2 shows the adsorption isotherms for the briquettes. For the carbon tetrachloride, the isotherms for the briquette and loose powder were the same up to 0.37 cc/cc capacity, indicating that the same type of aggregates are present whether in loose powder or in the briquette. The void volume from 0.37 to 0.67 or 0.30 cc/cc is formed on briquetting. The isotherms also indicate that the volume of clos-

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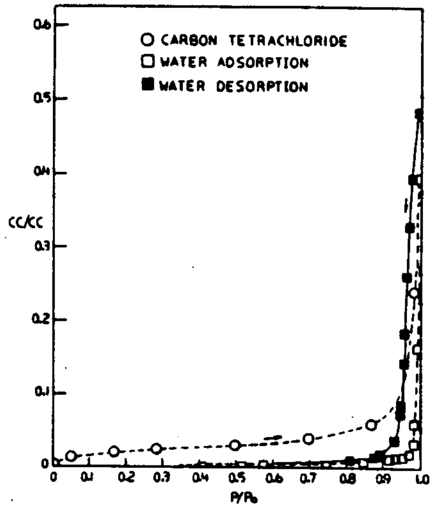

Figure 2. Adsorption isotherms on briquettes.

est hexagonally packed aggregates constitute about 10% of the void volume and surface area. If they consist of 12 particles each, they would be numerous and could be the nuclei for the larger aggregates. The isotherms also indicate that the distance between aggregates is very large. The mean distance between the particles (260 A dia) is about 180 A. This is based on calculated surface area of 76 m^2/cc and 0.67 cc/cc void volume. For the larger aggregates, the range is from 1000 to 100,000 A. In the closest hexagonally packed aggregates, the mean distance is about 29 A.

Collapse Strength Tests

Figures 4 and 3 show the results of the collapse strength tests for carbon tetrachloride and water, respectively. The collapse strength starts to increase immediately on addition of adsorbate. For carbon tetrachloride at 0.034 cc/cc content, corresponding to the BET theoretical surface area coverage of 68 m^2/cc, there is a definite strength increase. For both adsorbates, the strength increases to 0.35 cc/cc adsorbate content and then decreases again. When the void space is filled at 0.67 cc/cc, the briquette becomes plastic; it does not collapse but slowly deforms. On desorption, there is considerable hysteresis. The maximum is reached at about 0.28 cc/cc adsorbate content and returns to a strength level five times greater than the original.

Discussion and Conclusions

The briquette is made up of aggregates of which the smallest and most densely packed are made of the 260A diameter particles closest hexagonally packed. These aggregates provide only 0.07 cc/cc or 10% of the void volume and 10% of the surface area, but are numerous enough to act as nuclei for the larger aggregates. The most significant characteristic is that they also have smallest average interparticulate distance, 29 A average. The interparticulate distance for the remainder of the aggregates which accounts for 90% of the void volume and surface area, vary from about 60 to 100,000 A.

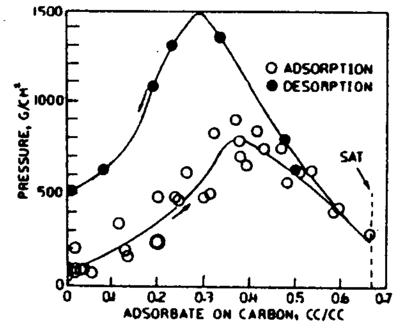

Figure 3. Collapse pressure as function of water content.

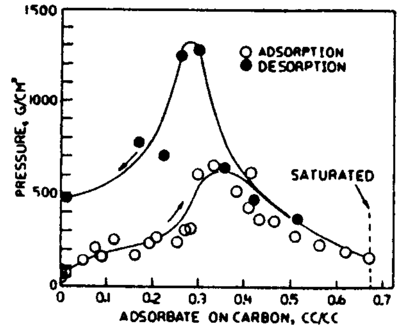

Figure 4. Collapse pressure as function of carbon tetrachloride content.

If adsorption for carbon tetrachloride, proceeds according to the BET multilayer adsorption theory, there would be no significant steric hindrance; that is, opposite wall attraction, on completion of the first layer, and consequently, no strengthening of the briquette. But at the required liquid volume, 0.034 cc/cc, to complete the first layer, there is a significant increase of briquette strength from 70 to 120 g/cm^2 required collapse pressure. Ninety percent of the 0.034 cc/cc would be outside of small dense aggregates. On completion of the second layer, or liquid content of 0.68 cc/cc, there would still be a very insignificant opposite wall attraction, but the briquette strength increased to 180 g/cm^2 collapse pressure. It is now apparent, further adsorption in the larger interparticulate or interaggregate void volume would have even a lesser possibility of increasing the briquette strength. If the carbon tetrachloride is adsorbed by capillary condensation, carbon tetrachloride molecules bridge the gap between the particulate and aggregate surfaces and draw them toward each other, thereby, strengthening the briquette as the amount of carbon tetrachloride is increased. The same occurs with water except adsorption starts at a higher relative vapor pressure than for the carbon tetrachloride.

The variations in briquette strength is a definite indication that capillary condensation is the primary mode of adsorption over the entire relative pressure range.