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APPENDICES

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Appendix A

Table A.1 Properties of commercial activated carbons (TIS 900-1989).

Table A.2 Properties of commercial powdered activated carbon.

Table A.3 Properties of commercial granular activated carbon.



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Table A.1 Properties of commercial activated carbons (TIS 900-1989).

Types	Size	%Moisture	BD(g/cm ³)	IA(mg/g)
Powdered activated carbon	< .15 mm	-	0.20-0.75	> 600
Granular activated carbon	> .15 mm	< 8	>0.36	> 600

Table A.2 Properties of commercial powdered activated carbon.

Trade names	Raw material	Ash (%)	BD (g/cm ³)	S _{BET} (m ² /g)	MB (mg/g)	IA (mg/g)
UDP CHEMICAL (Thailand)						
MDPI1-7325	Coconut shel	3	0.40-0.55	1200-1300	230	1050
PN3	-	8	0.46	-	-	800
Taiko PKW	Sawdust	2	-	-	-	-
Shirasagi KW	Sawdust	3	-	-	-	-
Nuchar SA	-	3-5	0.37-0.40	-	-	900
Nuchar SN	-	3-5	0.37-0.40	-	-	900
PICA U.S.A. INC.						
P1400	Wood	7	-	-	-	1200-1300
GX203	Coconut shell	7	-	-	-	1100-1200
GX213	Coconut shell	7	-	-	-	900-1100
R160	Coconut shell	7	-	-	-	1500
CARBON LINK LIMITED						
MesocarbFGT	Coal based	-	0.45	900	-	850
MesocarbFGT	Coal based	-	0.55	550	-	500

Table A.3 Properties of commercial granular activated carbon.

Trade names	Raw material	Ash (%)	BD (g/cm ³)	S _{BET} (m ² /g)	MB (mg/g)	IA (mg/g)
UDP CHEMICAL (Thailand)						
MD-G 7746	Coconut shell	3	0.40-0.45	1100-1200	200	1050
MD-W 7830	Coconut shell	3	0.50-0.55	1150-1250	220	1050
PICA U.S.A. INC.						
G 201	Coconut shell	3	0.48-0.53	1100-1200	-	-
G 202	Coconut shell	5	0.41-0.47	1250-1400	-	-
TA70	Coconut shell	5	0.48-0.52	1250-1300	-	-
G 411	Wood Based	6	0.25-0.31	600-700	-	-
GX 191	Wood Based	10	0.20	1000-1100	-	-
G 352	Coal Based	6	0.37-0.42	-	-	-
CARBON LINK LIMITED						
Microcarb TH5	-	1	0.53	900	-	-
Microcarb FY5	-	2	0.49	1150	-	1100
Microcarb AY5	-	0.5	0.49	1150	-	1100
Mesocarb BKK100	-	0.41	0.50	1050	-	1000
Mesocarb C40	-	6	0.40	1150	-	1100
Mesocarb D43	-	5	0.43	1100	-	1050
ROOLWORTH INDUSTRIES (Malaysia)						
-	Palm-oil shell	5	-	-	-	900-1000
LURGI						
Hydraffin N 20	-	-	0.48	950	-	900
PJAC (Japan)						
CC-4X10	Coconut shell	5	0.42-0.47	1200-1300	170	1050
CG- 6X8	Coconut shell	5	0.42-0.47	1200-1300	170	1100
CW-8X30	Coconut shell	5	0.45-0.50	1050-1150	170	1050
SYBRON	Bituminous	-	0.50-0.55	900	220	900

Appendix B

- Appendix B.1** Standard Practice for Proximate Analysis of Coal and Coke (ASTM D 3172-89).
- Appendix B.2** Standard Test Method for Moisture in the Analysis Sample of Coal and Coke (ASTM D 3173-87).
- Appendix B.3** Standard test Method for Ash in the Analysis Sample of Coal and Coke from Coal (ASTM D 3174-93).
- Appendix B.4** Standard test Method for Volatile Matter in the Analysis Sample of Coal and Coke (ASTM D 3175-89a).
- Appendix B.5** Standard Test Method for Determination of Iodine Number of Activated Carbon (ASTM D 4607-94).
- Appendix B.6** Standard testing Method of Methylene blue number of Activated Carbon (JIS K 1470-1991).
- Appendix B.7** Standard Test Method for Apparent Density of Activated Carbon (ASTM D 2884-89).
- Appendix B.8** Standard Test Methods for Carbon Black- Surface area by multipoint B.E.T. Nitrogen Adsorption (ASTM D 4820-96a).
- Appendix B.9** Standard Test Method for Chemical analysis of Wood Charcoal (ASTM D.1762-84)

Appendix B.1

Standard Practice for Proximate Analysis of Coal and Coke (ASTM D 3172-89)

This practice covers the determination of moisture, volatile matter, and ash and the calculation of fixed carbon on coals and cokes sampled and prepared by prescribed methods and analyzed according to ASTM established procedures.

Test methods, as herein described, can be used to establish the rank of coals, show the ratio of combustible to incombustible constituents, provide the basis for buying and selling, and evaluate for beneficiation or for other purposes.

Test Methods

1. *Moisture* - Test Method D 3173.
2. *Ash* - Test Method D 3174.
3. *Volatile Matter* - Test Method D 3175.
4. *Fixed Carbon* - The fixed carbon is a calculated value. It is the resultant of the summation of percentage moisture, ash, and volatile matter subtracted from 100. All percentages shall be on the same moisture reference base.

$$\text{Fixed carbon, \%} = 100 - (\text{moisture, \%} + \text{ash, \%} + \text{volatile matter, \%})$$

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Appendix B.2

Standard Test Method for Moisture in the Analysis Sample of Coal and Coke (ASTM D 3173-87)

This test method covers the determination of moisture in the analysis sample of coal or coke. It is used for calculating other analytical results to a dry basis. When used in conjunction with the air drying loss as determined in accordance with Method D 2013 or Method D 346, each analytical result can be calculated to an as-received basis.

Moisture is determined by establishing the loss in weight of the sample when heated under rigidly controlled conditions of temperature, time and atmosphere, sample weight, and equipment specifications.

Analysis Sample

The analysis sample is that sample which has been pulverized to pass 250 μm (No. 60) sieve as prepared in accordance with Method D 346 or Method D 2013.

Procedure for Sample Passing a 250 μm (No. 60) Sieve

1. Heat the empty capsules under the conditions at which the sample is to be dried, place the stopper or cover on the capsule, cool over a desiccant for 15 to 30 min, and weigh. Dip out with a spoon or spatula from the sample bottle approximately 1 g of the sample. Put this quickly into the capsule, close, and weigh at once to the nearest ± 0.1 mg.

2. An alternative procedure for weighing the sample (more subject to error) is as follows. After transferring an amount of the sample slightly in excess of 1 g bring to exactly 1g in weight (± 0.5 mg) by quickly removing the excess weight of the sample with a spatula. The utmost dispatch must be used in order to minimize the exposure of the sample until the weight is determined.

3. After removing the covers, quickly place the capsules in a preheated oven (at 104 to 110°C) through which passes a current of dry air. (The current of dry air is not necessary for coke.) Close the oven at once and heat for 1 hr. Open the oven, cover the capsules quickly, cool in a desiccator over desiccant, and weigh as soon as the capsules have reached room temperature.

4. Use the percentage of moisture in the sample passing a 250 μm (No. 60) sieve to calculate the results of the other analysis to a dry basis

Calculations

Calculate the percent moisture in the analysis sample as follow:

$$\text{Moisture in analysis sample, \%} = [(A - B) / A] \times 100$$

where:

A = grams of sample used, and

B = grams of sample after heating.

Appendix B.3

Standard Test Method for Ash in the Analysis Sample of Coal and Coke from Coal (ASTM D 3174 – 93)

This test method covers the determination of the inorganic residue as ash in the analysis sample of coal or coke as prepared in accordance with Method D 2013 or Practice D 346. The results obtained can be applied as the ash in the proximate analysis, Practice D 3172.

Ash is determined by weighing the residue remaining after burning the coal or coke under rigidly controlled conditions of sample weight, temperature, time, atmosphere, and equipment specifications.

Procedure

1. The sample shall be the material pulverized to pass No. 60 (250 μm) sieve.
2. Transfer approximately 1 g (weighed to the nearest 0.1 mg) of the thoroughly mixed sample to a weighed capsule and cover quickly. An alternative way is to use the dried coal from the moisture determination in Test Method D 3173. Place the capsule containing the sample in a cold furnace and heat gradually at such a rate that the temperature reaches 450 to 500°C in 1 hr.

3. Continue heating so that a temperature of 700 to 750°C is reached by the end of the second hour. Continue the ashing at 700 to 750°C for two additional hours (*Note 1*). Remove the capsule from the muffle, place the cover on the capsule, cool under conditions to minimize moisture pickup, and weigh.

Note 1. While the 4 hr incineration interval described is sufficient with most coals to reach a condition of complete burn-off, certain cokes and nonreactive coals may require additional time. If unburned carbon particles are observed, or if duplicate results are suspect, the samples should be returned to the furnace for sufficient time to

reach a constant weight (± 0.001 g). By this means pyritic sulfur will be oxidized and expelled before the calcite is decomposed. An ample supply of air in the muffle “2 to 4 changes per minute” must be assured at all times to ensure complete oxidation of the pyritic sulfur and to remove the SO_2 formed. The 4 hr time limit may be reduced if the sample reaches a constant weight at 700 to 750°C in less than 4 hr.

Calculation

Calculate the ash percent in the analysis sample as follows:

$$\text{Ash in analysis sample, \%} = [(A - B) / C] \times 100$$

where:

A = weight of capsule, cover, and ash residue, g,

B = weight of empty capsule and cover, g, and

C = weight of analysis sample used, g.

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Appendix B.4

Standard Test Method for Volatile Matter in the Analysis Sample of Coal and Coke (ASTM D 3175 - 89a)

This test method determines the percentage of gaseous products, exclusive of moisture vapor, in the analysis sample which are released under the specific conditions of the test.

Volatile matter is determined by establishing the loss in weight resulting from heating a coal or coke under rigidly controlled conditions. The measured weight loss, corrected for moisture as determined in Test Method D 3173 establishes the volatile matter content. Two procedures are described to permit conformity with differences in sample behavior.

Procedure

1. The sample shall be the material pulverized to No. 60 (250 μm) sieve in accordance with Method D 2013 or Method D 346.

2. Procedure for Nonsparking Coals and Cokes:

2.1 Weigh 1 g of the sample in a weighed platinum crucible, close with a cover which fits closely enough so that the carbon deposit from bituminous, subbituminous, and lignite coals does not burn away from the underside, place on platinum or Nichrome-wire supports and insert directly into the furnace chamber, which shall be maintained at a temperature of $950 \pm 20^\circ\text{C}$, and lower immediately to the 950°C zone. Regulation of the temperature to within the prescribed limits is critical. After the more rapid discharge of volatile matter has subsided, as shown by the disappearance of the luminous flame, or in the case of coke, after 2 or 3 min, inspect the crucible (see *Note 1*) to verify that the lid is still properly sealed. If necessary, reseal the lid to guard against the admission of air into the crucible. Do this as rapidly as possible by raising the crucible to the top of the furnace chamber, reposition the lid to more perfectly seal the crucible, then lower the

crucible immediately back to the 950°C zone. After heating for a total of exactly 7 min, remove the crucible from the furnace and without disturbing the cover, allow it to cool. Coke should be cooled in a desiccator. Weigh as soon as cold. The percentage loss of weight minus the percentage moisture equals the volatile matter. With some strongly caking low-volatile and medium-volatile bituminous coals, the coke button may be broken with explosive violence due to the liberation of volatile matter within the button. This is usually designated as popping. Such popping may blow the lid off the crucible and cause mechanical losses of the coked material. When such popping is observed, the determination shall be rejected and the test repeated until popping does not occur.

Note 1 Inspection of the crucible may be aided by the use of a mirror held above the furnace well.

3. Modified Procedure for All Sparking Fuels:

3.1 Fuels that do not cake or cake weakly when volatile matter is determined shall be watched closely for sparking during the heating period (*Note 2*): also, at the end of the test the crucible cover shall be inspected for ash deposits, and the presence of such deposits shall be considered as evidence of sparking.

3.2 All fuels that spark when the volatile matter is determined by the methods described in 1 shall be treated as follows: The sample shall be given a preliminary gradual heating such that a temperature of $600 \pm 50^\circ\text{C}$ is reached in 6 min (*Note 2*). After this preliminary heating the sample shall be heated for exactly 6 min at $950 \pm 20^\circ\text{C}$. If sparking is then observed, the determination shall be rejected and the test repeated until no sparking occurs either during the preliminary heating or during the 6 min period at 950 C. Remove the crucible from the furnace, cool on a metal cooling block, and weigh. To ensure uniformity of results, keep the cooling period constant and do not prolong beyond 15 min. The percentage loss in weight minus the percent moisture in accordance with Test Method D 3173, is the volatile matter. All analysis by this test method shall be so marked when reported to indicate that the modified procedure was used.

Note 2 If a tubular furnace of the Fieldner type is used for the determination of volatile matter, the preliminary gradual heating may be accomplished by moving the crucible to predetermined positions in the cooler top zone of the furnace. Due to variations in the heating characteristics of the furnace, the operator must predetermine by thermocouple the proper positions to meet a preliminary heating rate as specified in 3.2. A mechanical device to lower the crucible into the furnace may be used to facilitate control of the lowering operation.

Calculation

1. Calculate the weight loss percent as follows:

$$\text{Weight loss, \%} = [(A - B) / A] \times 100$$

where:

A = weight of sample used, g, and

B = weight of sample after heating, g.

2. Calculate the volatile matter percent in the analysis samples as follows:

$$\text{Volatile matter in analysis sample, \%} = C - D$$

where:

C = weight loss, %, and

D = moisture, %.

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Appendix B.5

Standard Test Method for Determination of Iodine Number of Activated Carbon (ASTM D 4607-94)

This test method covers the determination of the relative activation level of unused or reactivated carbons by adsorption of iodine from aqueous solution. The amount of iodine absorbed (in milligrams) by 1 g of carbon using test conditions listed herein is called the iodine number.

This test method is based upon a three-point adsorption isotherm. A standard iodine solution is treated with three different weights of activated carbon under specified conditions. The carbon treated solutions are filtered to separate the carbon from the treated iodine solution (filtrate). Iodine remaining in the filtrate is measured by titration. The amount of iodine removed per gram of carbon is determined for each carbon dosage and the resulting data used to plot an adsorption isotherm. The amount of iodine adsorbed (in milligrams) per gram of carbon at a residual iodine concentration of 0.02 N is reported as the iodine number.

1. Preparation of Solutions

1.1 Hydrochloric Acid Solution (5% by weight). Add 70 mL of concentrated hydrochloric acid to 550 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

1.2 Sodium Thiosulfate(0.100N). Dissolve 24.820 g of sodium thiosulfate in approximately 75 ± 25 mL of freshly boiled distilled water. Add 0.10 ± 0.01 g of sodium carbonate to minimize bacterial decomposition of the thiosulfate solution. Quantitatively transfer the mixture to a 1 L volumetric flask and dilute to the mark. Allow the solution to stand at least 4 days before standardizing. The solution should be stored in an amber bottle.

1.3 Standard Iodine Solution ($0.100 \pm 0.001 N$). Weigh 12.700 g of iodine and 19.100 g of potassium iodide (KI) into a beaker. Mix the dry iodine and potassium iodide. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increments of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the solution to stand a minimum of 4 hr to ensure that all crystals are thoroughly dissolved. Occasional stirring during this 4 hr period will aid in the dissolution. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water. It is important that the standard iodine solution has an iodide-to-iodine weight ratio of 1.5 to 1.0. Store the solution in an amber bottle.

1.4 Potassium Iodate Solution ($0.1000 N$). Dry 4 or more grams of primary standard grade potassium iodate (KIO_3) at $110 \pm 5 ^\circ C$ for 2 hr and cool to room temperature in a desiccator. Dissolve 3.5667 ± 0.1 mg of the dry potassium iodate in about 100 mL of distilled water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water. Mix thoroughly and store in a glass-stoppered bottle.

1.5 Starch Solution. Mix 1.0 ± 0.5 g of starch with 5 to 10 mL of cold water to make a paste. Add an additional 25 ± 5 mL of water while stirring to the starch paste. Pour the mixture, while stirring, into 1 L of boiling water and boil for 4 to 5 min. This solution should be made fresh daily.

2. Standardization of Solutions

2.1 Standardization of $0.100 N$ Sodium Thiosulfate. Pipet 25.0 mL of potassium iodate (KIO_3) solution from 1.4 into a 250 mL titration (or wide-mouthed Erlenmeyer) flask. Add 2.00 ± 0.01 g of potassium iodide (KI) to the flask and shake the flask to dissolve the potassium iodide crystals. Pipet 5.0 mL of concentrated hydrochloric acid into the flask. Titrated the free iodine with sodium thiosulfate solution until a light yellow color is observed in the flask. Add a few drops of starch indicator (1.5) and continue the titration dropwise until one drop produces a colorless solution. Determine sodium thiosulfate normality as follows:

$$N_1 = (P \cdot R) / S$$

where:

N_1 = sodium thiosulfate, N ,

P = potassium iodate, mL,

R = potassium iodate, N , and

S = sodium thiosulfate, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications should be done if the range of values exceeds 0.003 N .

2.2 Standardization of $0.100 \pm 0.001 N$ Iodine Solution. Pipet 25.0 mL of iodine solution (1.3) into a 250 mL wide mouthed Erlenmeyer flask. Tritrate with standardized sodium thiosulfate (2.1) until the iodine solution is a light yellow color. Add a few drops of starch indicator (1.5) and continue titration dropwise until one drop produces a colorless solution. Determine the iodine solution normality as follows:

$$N_2 = (S \cdot N_1) / I$$

where:

N_2 = iodine, N ,

S = sodium thiosulfate, mL,

N_1 = sodium thiosulfate, N , and

I = iodine, mL.

The titration step should be done in triplicate and the normality results averaged. Additional replications be done if the range of values exceeds 0.003 N . The iodine solution concentration must be $0.100 \pm 0.001 N$. If this requirement is not met, repeat 1.3 and 2.2.

3. Procedure

3.1 The procedure applies to either powdered or granular activated carbon. When granular carbon is to be tested, grind a representative sample of carbon until 60 wt % (or more will pass through a 325 mesh screen) and 95 wt % or more will

pass through a 100 mesh screen. Carbon received in the powdered form may need additional grinding to meet the particle size requirement given above.

3.2 Dry the ground carbon from 3.1 in accordance with Test Method D 2867. Cool the dry carbon to room temperature in a desiccator.

3.3 Determination of iodine number requires an estimation of three carbon dosages. Section 4 and Table B. 5.1 describes how to estimate the carbon dosages to be used. After estimating carbon dosages, weigh three appropriate amounts of dry carbon to the nearest milligram. Transfer each weighed sample of carbon to a clean, dry 250 mL Erlenmeyer flask equipped with a ground glass stopper.

3.4 Pipet 10.0 mL of 5 wt% hydrochloric acid solution into each flask containing carbon. Stopper each flask and swirl gently until the carbon is completely wetted. Loosen the stoppers to vent the flasks, place on a hot plate in a fume hood, and bring the contents to a boil. Allow to boil gently for 30 ± 2 s to remove any sulfur which may interfere with the test results, Remove the flasks from the hot plate and cool to room temperature.

3.5 Pipet 100.0 mL of 0.100 *N* iodine solution into each flask. Standardize the iodine solution just prior to use. Stagger the addition of iodine to the three flasks so that no delays are encountered in handling. Immediately stopper the flasks, and shake the contents vigorously for 3 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper (Whatman No. 2V or equivalent) into a beaker. Filtration equipment must be prepared in advance so no delay is encountered in filtering the samples.

3.6 For each filtrate, use the first 20 to 30 mL to rinse a pipet. Discard the rinse portions. Use clean beakers to collect the remaining filtrates. Mix each filtrate by swirling the beaker and pipet 50.0 mL of each filtrate into a clean 250 mL Erlenmeyer flask. Titrate each filtrate with standardize 0.100 *N* sodium thiosulfate solution until the solution is a pale yellow. Add 2 mL of the starch indicator solution and continue the titration with sodium thiosulfate until one drop produces a colorless solution. Record the volume of sodium thiosulfate used.

4. Calculation

4.1 The capacity of a carbon for any adsorbate is dependent upon the concentration of the adsorbate in solution. The concentrations of the standard iodine solution and filtrates must be specified or known. This is necessary to determine an appropriate carbon weight to produce final concentrations agreeing with the definition of iodine number. The amount of carbon sample to be used in the determination is governed by the activity of the carbon. If filtrate normalities (C) are not within the range of $0.008 N$ to $0.040 N$, repeat the procedure using different carbon weights.

4.2 Two calculations are required for each carbon dosage, as X/M and C .

4.2.1 To calculate the value of X/M , first derive the following values:

$$A = (N_2) (12693.0)$$

where:

N_2 = iodine, N (from 2.2).

$$B = (N_1) (126.93)$$

where:

N_1 = sodium thiosulfate, N (from 2.1).

$$DF = (I+H) / F$$

where:

DF = dilution factor,

I = iodine, mL (from 2.2),

H = 5% hydrochloric acid used, mL, and

F = filtrate, mL.

For example, if 10 mL of HCl and 50 mL of filtrate are used: $DF = (100 + 10) / 50 = 2.2$

4.2.1.1 Calculate the value of X/M as follows:

$$X/M = [A - (DF) (B) (S)] / M$$

where:

X/M = iodine absorbed per gram of carbon, mg/g,

S = sodium thiosulfate, mL, and

M = carbon used, g.

4.2.2 Calculate the value of C as follows:

$$C = (N_f \cdot S) / F$$

where:

C = residual filtrate, N ,

N_f = sodium thiosulfate, N , and

F = filtrate, mL.

4.3 Using logarithmic paper, plot X/M (as the ordinate) versus C (as the abscissa) for each of the three carbon dosages. Calculate the least squares fit for the three points and plot. The iodine number is the X/M value at a residual iodine concentration (C) of 0.02 N . The regression coefficient for the least squares fit should be greater than 0.995.

4.4 Carbon dosage may be estimated as follows:

$$M = [A - (DF) (C) (126.93) (50)] / E$$

where:

M = carbon, g,

A = $(N_2) (12693.0)$,

DF = dilution factor (see 4.2.1)

C = residual iodine, and

E = estimated iodine number of the carbon.

Three carbon dosages are calculated using three values of C (usually 0.01, 0.02, and 0.03).

Table B.5.1 Find M for calculation iodine number by using ASTM D 4607-86.

IA	M			IA	M		
	C = 0.01	C = 0.02	C = 0.03		C = 0.01	C = 0.02	C = 0.03
300	3.766	3.300	2.835	1550	0.729	0.639	0.549
350	3.228	2.829	2.430	1600	0.706	0.619	0.531
400	2.824	2.475	2.126	1650	0.684	0.600	0.515
450	2.510	2.200	1.890	1700	0.664	0.582	0.500
500	2.259	1.980	1.701	1750	0.645	0.566	0.486
550	2.054	1.800	1.546	1800	0.628	0.550	0.472
600	1.883	1.650	1.417	1850	0.610	0.535	0.460
650	1.738	1.523	1.308	1900	0.594	0.521	0.447
700	1.614	1.414	1.215	1950	0.579	0.508	0.436
750	1.506	1.320	1.134	2000	0.565	0.495	0.425
800	1.412	1.237	1.063	2050	0.551	0.483	0.415
850	1.329	1.164	1.000	2100	0.538	0.471	0.405
900	1.255	1.100	0.945	2150	0.525	0.460	0.396
950	1.189	1.042	0.895	2200	0.513	0.450	0.386
1000	1.130	0.990	0.850	2250	0.502	0.440	0.378
1050	1.076	0.943	0.810	2300	0.491	0.430	0.370
1100	1.027	0.900	0.773	2350	0.481	0.421	0.362
1150	0.982	0.861	0.739	2400	0.471	0.412	0.354
1200	0.941	0.825	0.709	2450	0.461	0.404	0.347
1250	0.904	0.792	0.680	2500	0.452	0.396	0.340
1300	0.869	0.761	0.654	2550	0.443	0.388	0.333
1350	0.837	0.733	0.630	2600	0.434	0.381	0.327
1400	0.807	0.707	0.607	2650	0.426	0.374	0.321
1450	0.779	0.683	0.586	2700	0.418	0.367	0.315
1500	0.753	0.660	0.567	2750	0.411	0.360	0.309

Appendix B.6

Standard testing method of Methylene Blue Number of Activated Carbon (JIS K 1470-1991)

1. Preparation of Solutions

1.1 Potassium dihydrogen phosphate solution. Dry of potassium dihydrogen phosphate (KH_2PO_4) in the oven at 110-120 °C for 2 hr and cool to room temperature in a desiccator. Dissolve 9.07 g of dry KH_2PO_4 with distilled water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water.

1.2 Disodium hydrogen phosphate solution. Dry of disodium hydrogen phosphate (Na_2HPO_4) in the oven at 110-120 °C for 2 hr and cool to room temperature in a desiccator. Dissolve 23.88 g of dry Na_2HPO_4 with distilled water. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with distilled water.

1.3 Buffer solution. Mix 400 mL of 1.1 and 600 mL of 1.2. The pH of this solution is approximately 7.

1.4 Standard methylene blue solution. Dry of methylene blue in the oven at 105 ± 5 °C for 4 hr and cool to room temperature in a desiccator. Dissolve 1.2 g of dry methylene blue with buffer solution. Quantitatively transfer to a 1 L volumetric flask and fill to the mark with buffer solution.

2. Procedure

2.1 Dry of activated carbon at 150 ± 5 °C (3 hr is usually sufficient).

2.2 Weigh the specified amount (*Note 1*) of activated carbon to the nearest 1 mg, transfer to an Erlenmeyer flask with ground-in stopper 100 mL, and add methylene blue solution 25 mL.

2.3 Shake at room temperature using a shaker for 30 min.

2.4 Take methylene blue solution 10 mL into one mark volumetric flask 50 mL, and add buffer solution up to the marked line. Further, take its 5 mL into other

one mark volumetric flask 500 mL, and add buffer solution up to the marked line. In this case the concentration of methylene blue solution is 0.24 mg/l.

2.5 Measure the absorbance at wavelength of 665 nm with contrasting to buffer solution.

Note 1 Weigh the sample by dividing into three stage degree so that the mass interval does not exceed 0.02 g corresponding to anticipated methylene blue adsorption performance to the sample 0.1 to 0.3 g.

3. Preparation of calibration curve

3.1 Take methylene blue solution 10 mL into one mark volumetric flask 50 mL, and add buffer solution up to the marked line. From this solution, take 5, 10, 25 and 50 mL into respective one mark volumetric flask 500 mL, and add buffer solution up to the marked line.

3.2 For these solution, prepare the relation curve between the concentration of methylene blue solution (0.24 to 2.4 mg/l) and the absorbance at 665 nm in wavelength and obtain from this the remaining concentration of methylene blue.

4. Calculation

Using the remaining concentration of methylene blue obtain in 3, the methylene blue amount (mg/g) shall be calculated using the following formula.

$$Q = \frac{(1200-C) (25/1000)}{S}$$

where:

Q = Methylene blue adsorption amount (mg/g),

C = remaining concentration of methylene blue (mg/l),

S = mass of activated carbon (g) and

1200 = concentration of methylene blue solution (mg/l).

5. Preparation of adsorption isotherm in methylene blue solution.

Plot the remaining concentration of methylene blue solution obtained in 3 on the abscissa and the adsorption amount of methylene blue obtained using the calculation method of 4 on the ordinate of both logarithm graph and prepare the adsorption isotherm. From the adsorption isotherm obtain the methylene blue amount (mg/g) of sample when the remaining concentration of methylene blue as the methylene blue adsorption performance is 0.24 mg/L.



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Appendix B.7

Standard Test Method for Apparent Density of Activated Carbon (ASTM D 2884-89)

This test method covers the determination of the apparent density (bulk density) of granular activated carbon. It is determined on granular sample by measuring the volume packed by a free fall from a vibrating feeder into a 10 mL graduated cylinder and weighing the known volume. For purposes of this test method, granular activated carbon is defined as a minimum of 90% being larger than 80 mesh.

Procedure

1. Dry an adequate sample of the carbon to be tested to constant weight at $150 \pm 5^\circ\text{C}$.
2. Carefully place a representative sample of the activated carbon into the feed funnel so that the material does not prematurely flow into the graduated cylinder.
3. Fill the cylinder at a uniform rate up to the 10 mL mark.
4. Transfer the contents from the cylinder to a balance pan and weigh to the nearest 0.1 g.

Calculation

Calculate the bulk density as follows:

$$\text{Bulk density, g/mL} = \text{weight of activated carbon}/10$$

Appendix B.8

Standard Test Methods for Carbon Black-Surface area by multipoint B.E.T. Nitrogen Adsorption (ASTM D 4820-96a)

A carbon sample is placed in a known volume cell and evacuated to less than 5 millitorr of vacuum. Using the ideal gas equation, the volume of nitrogen required to give a predetermined relative pressure is calculated and dosed into the sample cell. Any additional nitrogen required to attain this relative pressure is due to adsorption by the carbon. Based on the volume of nitrogen adsorbed at various relative pressures, the surface area is calculated.

Theory of Test Method

The determination of surface area from the B.E.T. theory is a straight forward application of the B.E.T. equation:

$$\frac{1}{V [(P_0/P) - 1]} = \frac{1}{V_M C} + \frac{C-1}{V_M C} \times \frac{P}{P_0}$$

where:

V = volume of nitrogen adsorbed, cm³,

P = pressure, kPa,

P₀ = saturation vapor pressure of nitrogen, kPa,

V_M = volume of nitrogen that covers one monomolecular layer, cm³, and

C = B.E.T. constant.

A plot of 1/V [(P₀/P) - 1] versus P/P₀ will usually yield a straight line in the range 0.05 to 0.35. Solving the B.E.T. equation for V_M gives:

$$V_M = \frac{1}{M + B}$$

where:

M = slope of straight line, and

B = y-intercept.

Sample Preparation

1. Place a clean sample tube on the degassing station and heat at 150°C for 0.5 hr at a pressure below 2.7 Pa (0.02 mmHg). The insertion of a glass rod into the sample tube stem will reduce the void volume of the stem, thus improving testing precision.

2. Cool the sample tube to room temperature, then backfill, preferably with helium to atmospheric pressure. Place a stopper on the sample tube and weigh it to the nearest 0.0001 g and record as mass A .

3. For samples of unknown surface area, place approximately 0.1 g of carbon in the sample tube clean the sample tube stem with a pipe cleaner.

4. Place the sample tube containing the carbon on the degassing station and open the vacuum valve.

5. Place the heating mantle on the sample cell and heat at 150°C for 1 hr or longer to obtain and hold a pressure less than 2.7 Pa (0.02 mmHg).

6. Remove the heating mantle and allow the sample cell to cool to room temperature. Backfill the sample tube with helium to atmospheric pressure, remove from the degassing station, and close it with a stopper.

Sample Analysis

1. Place the sample tube containing the carbon on the nitrogen surface area analyzer.

2. Initial the experiment. The following experimental steps, necessary to attain accurate results, can be automatically determined by most automated nitrogen surface area analyzers.

2.1 *Leak Test*. The vacuum volumetric technique relies on precise pressure measurements to determine the surface area of carbon. Any leaks in the system will result in erroneous surface area measurements. A minimum of a 2 min leak test

of the sample and P_0 cells is required. Any significant pressure change in the cells over this time interval will result in the absorption of the experiment.

2.2 Transducer Linearization. If separate sample and transducer manifolds are utilized by the testing equipment, it is necessary to zero and linearize the transducers prior to each experiment.

2.3 Sample Cell Volume. In determining the void volume of the sample cell, both the warm zone and cold zone (section submerged in liquid nitrogen) must be measured prior to each analysis. Non-ideality gas corrections are applied to the volume of gas in the cold zone.

2.4 Saturated Vapor Pressure (P_0). The saturated vapor pressure of nitrogen is affected by the purity of the liquid nitrogen and ambient pressure. Dissolved impurities in the liquid N_2 will usually cause the bath temperature to increase sufficiently to cause a 1.4 to 2.7 kPa (10 to 20 mmHg) increase in theoretical vapor pressure. Often, P_0 is assumed to be 103 kPa (775 mmHg); however, for maximum accuracy the actual P_0 should be measured.

3. Once the above conditions are measured, volumes of pure nitrogen are calculated, measured, and dosed into the sample cell. This process is repeated until equilibrium conditions are satisfied at the desired relative pressure. The summation of the dose volumes required to reach a particular relative pressure constitute the adsorption volume data at the various relative pressures. The adsorption volumes are measured at relative pressures of 0.05 to 0.30. A minimum of 5 relative pressures are recommended.

4. Backfill the sample tube at ambient temperature with helium to atmospheric pressure. Remove the sample tube from the instrument, stopper it, and weigh it to the nearest 0.0001 g and record as mass B .

5. Nitrogen adsorbed by the carbon at the various relative pressures is used to prepare the B.E.T. plot. The data points that give the best straight line are used to calculate the slope and y-intercept. The slope and y-intercept are used to calculate the surface area. For examples of how to select the proper relative pressure range.

6. A B.E.T. plot that yields a negative y-intercept could be indicative of the presence of microprobes (< 3 nm diameter), but others factors can produce a negative y-intercept. The surface area is calculated from the pressure range that yields the highest correlation coefficient and positive y-intercept.

Calculation

1. Calculate the sample mass as follows:

$$S = B - A$$

where:

S = sample mass, g,

A = mass of sample tube, stem, and stopper, and

B = mass of sample tube, stem, stopper, and sample after analysis.

2. Calculate the nitrogen surface area (NSA) to the nearest $0.1 \times 10^3 \text{ m}^2/\text{kg}$ ($0.1 \text{ m}^2/\text{g}$) as follows:

$$\text{NSA} = \frac{V_M N A_{CS}}{22\,400 \times S}$$

where:

N = Avagadro's number, 6.023×10^{23} molecules/mole,

A_{CS} = Cross-section area of nitrogen molecule, $16.2 \times 10^{-20} \text{ m}^2/\text{molecule}$, and

22400 = number of cm^3 occupied by one mole of gas at STP.

Appendix B.9

Standard Test Method for Chemical analysis of Wood Charcoal (ASTM D 1762-84)

This test method covers the determination of moisture , volatile matter, and ash in charcoal made from wood. The sample is ground in a specified manner and the moisture determined as loss in weight in a drying oven at 105°C. Volatile matter is determined as loss in weight at 950°C under specified conditions. Ash is determined as the residue after burning to constant weight at 750°C.

Procedure (for Activated carbon Passing A No. 50 Sieve)

1. Make duplicated determination.

2. **Moisture** Heat the muffle furnace to 750°C and place previously ignited porcelain crucible (*Note 1*) and cover in the furnace for 10 min. Cool the crucibles in a desiccator for 1 hr. Weigh the crucibles and add to each approximately 1 g, weighed to the nearest 0.1 mg, of the ground sample. Place the samples in the oven at 105°C for 2 hr. Place the dried samples in a desiccator for 1 hr and weigh (*Note 2*).

Note 1 In practice, a crucible from a previous determination is used.

Note 2 The sample shall be considered oven-dry when, the decrease in weight of consecutive weightings is 0.0005g or less. Succeeding drying periods shall be not less than 1 hr.

3. **Volatile Matter** Heat the muffle furnace to 950°C. Preheat the crucibles used for the moisture determination, with lids in place and containing the sample, as follows: with the furnace door open, for 2 min on the outer ledge of the furnace (300°C) and then for 3 min on the edge of the furnace (500°C) (*Note 3*). Then move the samples to the rear of the furnace for 6 min with the muffle door closed. Watch the samples through a small peep-hole in the muffle door. If sparking occurs, results will be in error (*Note 4*). Cool the samples in a desiccator for 1 hr and weigh.

Note 3 Individual nichrome wire baskets to hold the crucibles are convenient.

Note 4 If the sparking sample does not check the results of its nonsparking duplicate within $\pm 0.5\%$, the analysis shall be repeated.

4.Ash Place the lids and the uncovered crucible used for the volatile matter determination, and containing the sample in the muffle furnace at 750°C for 6 hr. Cool the crucibles with lids in place in a desiccator for 1 hr and weigh. Repeat burning of the sample until a succeeding 1 hr period of heating results in a loss of less than 0.0005 g.

Calculation

1. Calculate the percentage of moisture in the sample as follows:

Calculate the moisture content as follows:

$$\text{Moisture, \%} = [(A-B)/A] \times 100$$

where:

A = grams of air-dry sample used, and

B = grams of sample after drying at 105°C .

2. Calculate the percentage of volatile matter content in the sample as follows:

$$\text{Volatile matter, \%} = [(B-C)/B] \times 100$$

where:

C = grams of sample after drying at 950°C .

3. Calculate the percentage of the sample as follows:

$$\text{Ash, \%} = (D/B) \times 100$$

where:

D = grams of residue.

Appendix C

Figure C.1 Calibration curve for determine zinc in anthracite activated carbon samples.

Figure C.2 Calibration curve for determine zinc in palm-oil shell activated carbon samples.

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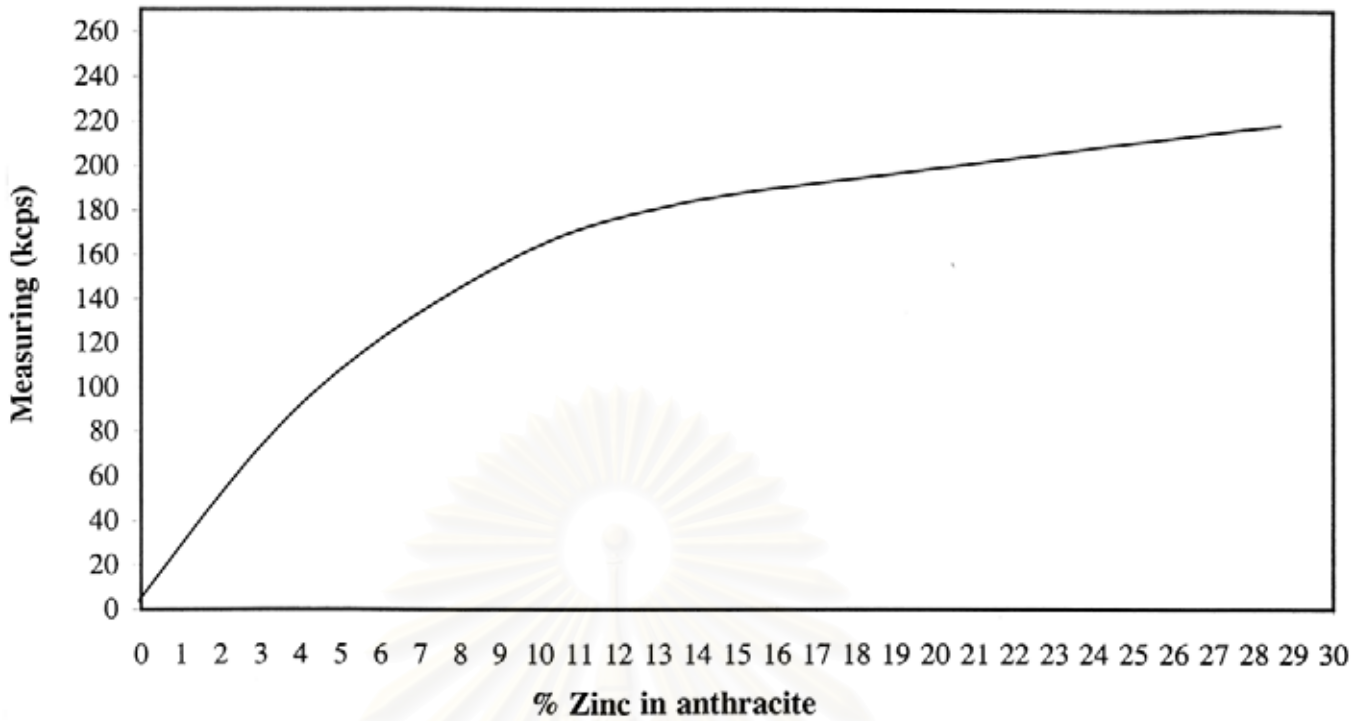


Figure C.1 Calibration curve for determine zinc in anthracite activated carbon samples.

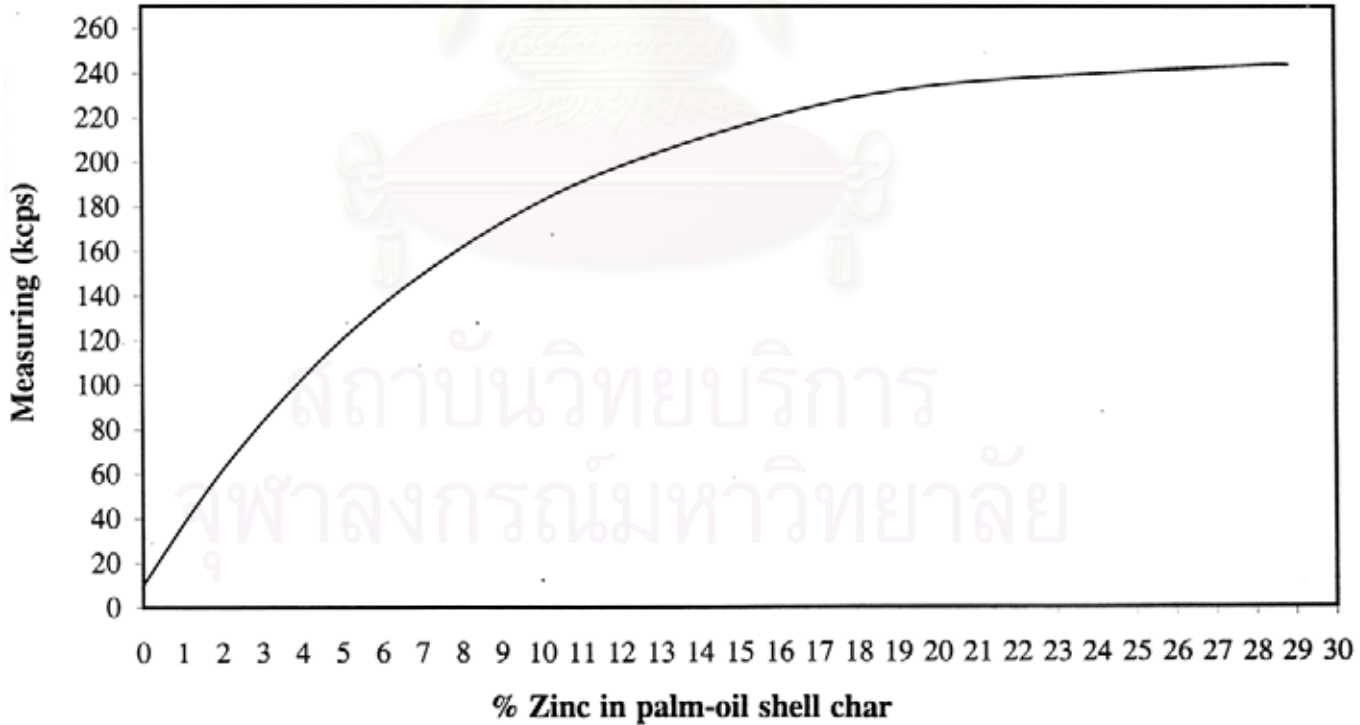


Figure C.2 Calibration curve for determine zinc in palm-oil shell activated carbon samples.

VITA

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