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APPENDICES

Appendix A.1

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 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 duplicate determinations.

2. **Moisture:** Heat the muffle furnace to 750°C and place previously ignited porcelain crucible *(Note1)* and covers 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 weight (note2).

Note1 In practice, a crucible from a previous determination is used

Note2 The sample shall be considered oven-dry when, the decrease in weight of consecutive weightings is 0.0005 g or less. Succeeding dry periods shall be not less is than 1 h.

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 oven, 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) *(Note3)*. Then move the samples to the rare 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 *(Note4)*. Cool the samples in a desiccator for 1 h and weigh.

Note3 Individual nichrome wire baskets to hold the crucibles are convenient.

Note4 If the speaking sample doses not check the results of its nonsparking duplicate within ±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:

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

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 ash in the sample as follows:

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

where: D = grams of residue.

Appendix A.2

Standard Test Method for Apparent Density of activated Carbon (ASTM D 1884-89)

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± 5°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 10 ml mark.
4. Transfer the contents from the cylinder to a balance pan and weight to the nearest 0.1 g.

Calculation

Calculate the bulk density as follows:

$$\text{Bulk density, g ml}^{-1} = \text{weight of activated carbon}/10$$

Appendix A.3

Standard Test Method for Moisture in Activated Carbon (ASTM D 2867-95)

These test methods for the determination of the moisture content of activated carbon, for this work used the oven-drying method. The oven-drying method is used when water is the only volatile material present and is in significant quantities, and the activated carbon is not heat-sensitive (some activated carbons can ignite spontaneously at temperatures as low as 150°C). A sample of carbon is put into dry, closed capsule (of know weight) and weighed accurately. The capsule is opened and placed with the lid in a preheated oven. The sample is dried to constant weight then removed from the oven and with the capsule closed, cooled to ambient temperature. The closed capsule is weighed again accurately. The weight loss expressed as a percentage of the weight of the original sample.

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

1. Dip out with a spoon or spatula from the sample bottle a 1 to 2 g representation sample. Put this into a predried tared capsule with lid, close and weight at once to the nearest 0.5 mg. The depth of the carbon in the capsule must not exceed 1.25 cm.

2. Remove the cover and place the capsule and cover in a preheated forced circulation oven (at 145 to 155°C). Close the oven and dry to constant weigh (3 hr normality sufficient). Open the oven and cover the capsules quickly. Cool in desiccator to ambient temperature and weigh.

Calculation

Calculate the moisture content as follows:

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

where: B = weight of capsule with cover, g

C = weight of capsule with cover plus original sample, g

D = weight of capsule with cover plus dried sample, g.

Appendix A.4

Standard Test Method for Total Ash content of Activated Carbon (ASTM D 2866-94)

This test method describes a procedure for the determination of total ash content of activated carbon. An accurately weighed sample of dried activated carbon is placed in a controlled-temperature muffle furnace for a period of several hours. When content weight has been achieved, the crucible is cooled to ambient temperature in a percentage of the weight of the original carbon sample.

Procedure

1. Ignite the crucible in the muffle furnace at 650 ± 25°C for 1 h Place the crucible in the desiccator. Cool to room temperature and weight to the nearest 0.1 mg.

2. Dry an adequate sample of activated carbon to constant weight at 150 ± 5°C (3 hr is usually sufficient).

3. Weigh out to the nearest 0.1 mg. sufficient dried activated carbon, so that the estimated amount of ash will be 0.1 g, into the ignited crucible and place the crucible in the furnace at 650 ± 25°C. Ashing will require from 3 to 16 hr, depending on the size and type of activated carbon. Ashing can be considered complete when constant weight is achieved.

4. Place the crucible in the desiccator and allow to cool to room temperature. After the simple has cooled in the desiccator, admit air slowly to avoid loss of ash from the crucible. Weigh to the nearest 0.1 mg.

Calculation

Calculate the ash content as follows:

$$\text{Total ash, \%} = [(D - B)]/(C - B)] \times 100$$

where: B = weight of crucible, g

C = weight of crucible plus original sample, g

D = weight of crucible plus ashed sample, g.

Appendix A.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 of 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 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 hydrocholic acid to 500 mL of distilled water and mix well. A graduated cylinder may be used for measurement of volume.

1.2 *Sodium Thiosulfate* (0.100 N). 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.

1.3 *Standard Iodine Solution* (0.100 ± 0.001 N). Weigh 12.700 g of iodine and 19.100 g of potassium iodine (KI) into a beaker. Mix the dry iodine and potassium iodine. Add 2 to 5 mL of water to the beaker and stir well. Continue adding small increment of water (approximately 5 mL each) while stirring until the total volume is 50 to 60 mL. Allow the solution into stand a minimum of 4 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 iodine-to-iodine weight ratio of 1.5 to 1. Store the solution in an amber bottle.

1.4 *Potassium iodate solution* (0.1000 N). Dry 4 or more grams of primary standard grad potassium iodate (KIO_3) at 110 ± 5 °C for 2 h 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 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 ± 0.5 g of water while stirring to the starch paste. Pour the mixture, white stirring, into 1 L of boiling water add 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 1.2 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:

$$N_1 = (P*R)/S$$

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 implications should be done if the range of values exceeds 0.003 *N*.

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

$$N_2 = (S * N,)/ I$$

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 should be done if the range of values exceed 0.003 *N*. The iodine solution concentration must be *0.100 ±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 unfit 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 from 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 iodine number requires an estimation of three carbon dosages. Section 4 and Table B.5.1 describes how to estimate the carbon dosages to use. After estimating carbon dosages, weight three appropriate amounts of dry carbon 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 whit the test results. Remove the flasks from hot plate and cool to room temperature.

3.5 Pipet 100.0 mL of 0.100 *N* iodine solution into each flask. Standardize the 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 30 ± 1 s. Quickly filter each mixture by gravity through one sheet of folded filter paper 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 portion. 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 standardized 0.100 *N* sodium thiosulfate solution units 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 carbon for any adsorbate is dependent upon the concentration of the adsorbate in solution. The concentrations of the standard iodine solution and filtrate 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, N

M = carbon used, g.

4.2.2 Calculate the value of C as follows:

$$C = (N_1 S) I F$$

where: C = residual filtrate, N

N_1 = sodium fhiosulfafe, mL

F = filtrate, mL.

4.3 Using logarithm c 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 grater 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_1) (12693.0)

DF = dilution factor (see 4.2.1)

C = residual iodine

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 A.5.1 M values for the calculation of iodine number by ASTM D 4607-86.

	M				M		
E	C=0.01	C=0.02	C=0.03	E	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	0.528	0.500
500	2.259	1.1980	1.701	1750	0.645	0.566	0.486
550	2.054	1.1800	1.546	1800	0.628	0.550	0.472
600	1.883	1.1650	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	0.388
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	0.896	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 A.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 desicator. Dissolve 9.07 g of KH_2PO_4 dry 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 desicator. 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. 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 desicator. 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 160 ± 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 fine. 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 Weight the sample by dividing into three stage degree so that the mass interval dose 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 solutions, 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 obtained in 3, the methylene blue amount (mg/g) shall be calculated using the following formula.

$$Q = (1200-C)(25/1000)/S$$

where: Q = Methylene blue adsorption amount (mg/g)

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

S = mass of activated carbon (g)

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

Appendix B

Paper Publications (Research Articles)

1. **Patnukao, P.**, and Pavasant, P. Activated carbon from *Eucalyptus camaldulensis Dehn* bark using phosphoric acid activation. Bioresource Technology. In press.
2. **Patnukao, P.**, Kongsuwan, A., and Pavasant, P. Batch studies of adsorption of copper and lead on activated carbon from *Eucalyptus camaldulensis Dehn* bark. Journal of Environmental Science. In press.
3. Kongsuwan, A., **Patnukao, P.**, and Pavasant, P. Binary component sorption of Cu(II) and Pb(II) with activated carbon from *Eucalyptus camaldulensis Dehn* bark. Submitted to Journal of Industrial and Engineering Chemistry.
4. **Patnukao, P.**, and Pavasant, P. Adsorption of copper and lead on activated carbon from *Eucalyptus camaldulensis Dehn* bark: column studies. In process.
5. **Patnukao, P.**, and Pavasant, P. Desorption of Cu(II) and Pb(II) from activated carbon by solvent exaction. In process.

Appendix C

Paper Publications (Conference Articles)

1. Kongsuwan, A., **Patnukao, P.**, and Pavasant, P. 2006. Removal of Metal Ion from Synthetic Waste Water by Activated Carbon from *Eucalyptus camaldulensis Dehn* Bark. JGSEE and Kyoto University 2nd Joint International Conference on: 21 - 23 Nov. 2006, Nai Lert Park Hotel, Bangkok.
2. **Patnukao, P.**, and Pavasant, P. 2006. Preparation of Granular Activated Carbon from Eucalyptus Bark for the Sorption of Cu^{2+} and Pb^{2+}. The Regional symposium on Chemical Engineering (RSCE 2006), 3-5 December 2006, Nanyang Executive Center, Singapore.

BIOGRAPHY

Miss Phussadee Patnukao was born on 27th December, 1979 in Trat. She finished her higher secondary courses from Trattakarnkhun school (Trat) in march, 1998. After that, she studied in the Department of General Science in Faculty of Science at Chulalongkorn University and graduated her Bachelor degree in 2002. She began her further study for Master's degree in Inter-department of Environment Science in Graduate School at Chulalongkorn University and received her Master's degree in April 2004. In the same year, she continued her Ph.D. study in International Environmental Management at Chulalongkorn University. During her doctoral learning, she was granted from the 90th Anniversary of Chulalongkorn University and joined the 13th Regional Symposium on Chemical Engineering at Nanyang Technological University, Singapore. She achived her Ph.D. degree in February 2008.