Chapter 34

CIP CARBONS - SELECTION, TESTING AND PLANT MONITORING

Dr. Jim Avraamides

Mines Department of Western Australia
Chief, Mineral Processing Laboratory
Chemistry Centre, WA

The properties of activated carbon which are important for gold recovery applications include activity, capacity, wear resistance, particle size distribution and bulk density. There are experimental difficulties in attempting to simulate the plant environment in the laboratory in order to measure the above properties. However, standard tests can be set up to compare the characteristics of different carbons and to assist in monitoring the performance of carbon in CIP operations. These laboratory tests are critically evaluated and data are presented on the performance of carbon from a number of CIP plants.

REVIEW OF TEST METHODS

The rapid expansion of the gold industry has seen a corresponding increase in the use of activated carbon for gold (and silver) recovery from cyanide pulps and clear heap leach solutions. At present there are two types of activated carbon used widely for gold adsorption - namely coconut shell and coal or peat based extrudates. Activated carbons derived from coconut shell char are produced by several large and many more smaller manufacturers. The quality and the price are generally directly related. However, in recent years there has been an increased awareness of the necessity to purchase the most cost effective product. In response to this some simple tests were developed in the Mines Department to measure those carbon properties which the gold industry considered most important, ie kinetic activity and resistance to attrition.

There are many tests available for measuring the properties of activated carbon - a material that has a long history of applications in water treatment, gas adsorption, solvent recovery and food purification. Although the published, standard tests for activated carbon do have some relevance they are not specific for gold recovery carbons. Most activated carbon manufacturers have their own in-house methods for testing carbons and normally provide comprehensive data sheets for their customers. Unfortunately test methods vary between manufacturers so little benefit can be gained by comparing data sheets.

Most activated carbon data sheets include most of the following measurements:

a. Particle size range.

b. Surface area - by nitrogen absorption

c. Methylene blue, Iodine, or Carbon Tetrachloride (CTC) number.

d. Abrasion resistance or impact strength.

e. Kinetic activity.

f. Gold loading capacity.

An excellent discussion on the relationships (or lack of) among most of the above parameters was reported by Faulkner, Urbanic and Ruckel, 1987. It became apparent from the many requests from the Australian gold industry for carbon evaluation that the most sought after data were kinetic activity, particle size distribution, and wear characteristics. Over the last few years in conjunction with Murdoch University and Curtin University of Technology, and with the co-operation of the major distributors of activated carbon in Australia an extensive data base has been generated on the activity and wear properties of CIP carbons.

TEST METHODS

It should be emphasized from the start that the choice of test depends on whether the result is to be used for quality control in manufacture, plant design or comparison of carbons from different sources. Whereas measuring particle size distribution is a straight forward procedure using standard mesh sieves, other test methods such as kinetic activity (or gold loading rate) and abrasion or attrition resistance vary between laboratories. In particular, there seemed to be a tendency in all carbon manufacturer's data sheets for abrasion resistance values to lie above 99%!

It was decided at the beginning of this exercise to use two or three of the most successful (and highest priced) commercial carbons as reference materials. All measurements were then made using one or more of these as a control.

This was of particular importance for the activity test, the results of which were subject to changes in solution temperature, standard gold solution preparation and other experimental variables.

Activity Test

Several different methods for measuring the rate at which gold is adsorbed onto carbon from an alkaline cyanide solution have been proposed. There are differences in equipment (eg agitated baffled vessels, bottle rolls or flask shakers), solution composition, carbon concentration carbon particle size and carbon pre-treatment. For simplicity, speed and reliability the batch kinetic test developed and adapted by workers at Mintek in South Africa (Fleming and Nicol, 1984) and Murdoch University (Labrooy and Bax, 1985) was considered to be acceptable for the following reasons.

a) No special equipment was required apart from a bottle roll and a flame atomic absorption spectrophotometer (AAS).

b) Gold concentrations can be read directly on the AAS if in the range 0-10 ppm.

c) Time of the test is 3 hours and several samples are taken for gold analysis which smooths errors arising from sampling and measurement procedures.

d) Closely sized carbon samples are used in order to reduce the effects of external surface area differences between samples.

e) A control carbon is run with each batch of test carbons and the resulting activities are expressed either as a rate constant, k, or as a relative percentage activity.

The data obtained from the activity test only provide comparative activities. Results can be presented graphically as gold adsorption curves (Figure 1) or else used to calculate a rate constant, k, from the equation below which has been reported elsewhere in the literature (Labrooy and Bax, 1985).

$$\Delta[Au]_c^t = k[Au]_s^t\, t^n$$ which can be rewritten as

$$\log\ \Delta[Au]^t_{c/}[Au]^t_s = n \log\ t + \log k \quad ...(1)$$

where:

$\Delta[Au]_c^t$ = change in gold loading on the carbon since time, t = zero (ppm).

$[Au]_s^t$ = gold concentration in solution at time, t (ppm).

n = empirical constant dependent on carbon sample and having values in the range 0.6-1.5 and for fresh carbons around 1.0.

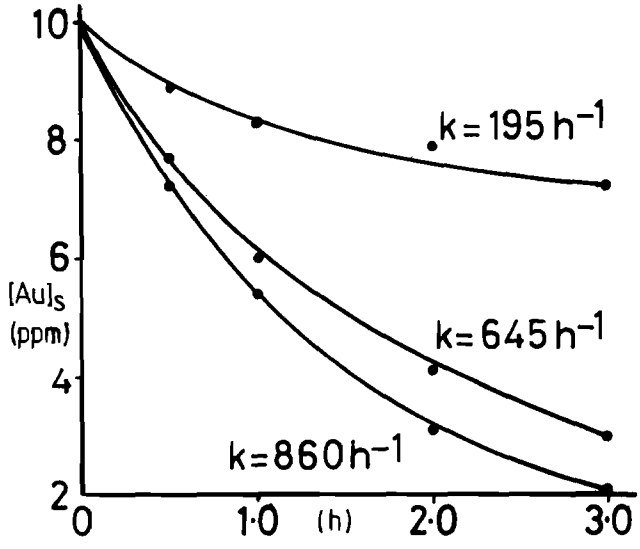

Figure 1. Activity data for coconut carbons

t = time of gold adsorption (h).

k = rate constant (h^{-1}, for $n = 1$)

Experimental conditions are as follows.

a) Weigh out 0.5 g screened carbon (dry weight basis)

b) Bottle roll at 100 rpm with 0.5 litres of a standard gold solution (10 ppm Au, 250 ppm NaCN, pH 10).

c) Take 5 mL samples at 0, 0.5, 1.0, 2.0, 3.0 hours and measure gold by flame AAS.

For coconut shell carbons the fraction 2.00-2.36 mm is normally taken. Extruded carbons are more regular in shape and size and are usually screened only at 1.00 mm to remove fines. High quality activated carbons have measured activity constants, k, in the range 700-1400 h^{-1} for temperatures between 15 and 35°C. The experimental variation in k values derived from the above test is about ±10% which is well within the scope of the method to rank carbon activities with confidence.

Relative activities are calculated from the gold adsorbed at time = 1 hour. This may be done graphically or can be calculated from the rate equation, which, at t = 1 hour simplifies to:

$$[Au]_s^{t=1} = 10{,}000/_{(k + 1000)} \quad ... (2)$$

Some values of k, solution gold concentrations and relative activities are given in Table 1. Equation 2 corrects for any variation in weight of carbon and initial concentration of gold. It is apparent from the data in Table 1 that there is a smaller incremental increase in relative activity than in k value. For the first two carbons in Table 1 the k values differ by a factor of about 7 whereas there is only a 4.5-fold increase in relative activity. Most clients prefer the

Table 1, Comparison between activity constants, k, and relative activities.

$k(h^{-1})$	$[Au]_S^{t=1}$ (ppm) Experimental	Calculated[1]	Relative Activity (%) (From Equn. 2)
114	9.1	9.0	17
817	5.7	5.5	76
1251	4.5	4.4	95
1465	4.1	4.1	100[2]

1) From k value using Equation 2.

2) Control sample arbitrarily set at 100%

relative activity data if they are seeking information on the merits of one carbon over another. At the upper end of the activity scale it can be seen that apparently large changes in k value translate into rather smaller increments in relative activity.

Assessment of fresh activated carbons is usually carried out using a distilled or deionized water solution of gold cyanide. Measurement of the activity of activated carbons from operating gold mines is preferably performed in process water. It has been established that carbons exposed to highly saline process waters exhibit diminished activities in low ionic strength test solutions. Consequently, requests are made to the client to provide sufficient process water if salinity levels are high (ie above 10,000 ppm). This is particularly significant in Western Australia where many CIP and heap leach operations are using ground waters containing up to 200,000 ppm total dissolved solids.

Wear Tests

Activated carbon is lost from CIP circuits through wet attrition in the abrasive agitated pulp environment of the adsorption circuit, during transport and screening operations and by oxidation or burning in the regeneration kiln. There will also be some losses through dry abrasion in the kiln. Several accelerated tests were devised or adapted from information available in the literature. All the tests involve subjecting the sized carbon sample to a wear regime and then measuring the proportion of undersize and oversize after screening at 1.00 mm. Carbons are always conditioned for a short time to remove imperfections and weak parts of the particles. This conditioning step often indicates how well the manufacturer has prepared the carbon for delivery. Some gold mining operaters condition all new carbon before it is sent to the adsorption circuit.

In the dry abrasion test a 50 g charge of dried sized carbon is pre-abraded for 10 minutes on a 1.00 mm standard brass sieve of 200 mm diameter using a 20 mm stainless steel ball and a "Ro-tap" sieve shaker. The +1.00 mm material (30 g) is then subjected to the same conditions for a further 60 minutes. The percentage ratio of oversize to total charge is the abrasion resistance [Equation (3)].

Table 2, Dry abrasion resistance for various activated carbons.

Sample	Particle Size (mm)	Dry abrasion resistance (percent)
A) Coconut shell	2.80-3.36	94.7
A) Coconut shell	2.00-2.36	93.7
A) Coconut shell	1.70-2.00	92.7
B) Coconut shell	2.00-2.36	85.0
C) Coconut shell	2.00-2.36	90.1
D) Extruded	+1.00 mm	96.5
E) Extruded	+1.00 mm	93.7

$$\text{Abrasion Resistance (in percent)} = \frac{\text{+1.00 mm oversize}}{\text{oversize + undersize}} \times 100 \qquad ...(3)$$

Abrasion resistances are related to particle size as shown by the data in Table 2.

Two wet attrition tests were devised - one using a water slurry of carbon and the second using an ore pulp. The equipment employed was a Wemco Attritioner comprising a baffled cylindrical vessel 160 mm diameter and 305 mm high and an electrically driven shaft at the bottom of which were attached 3 x 4 - bladed propellors, 34 mm apart, and covering an arc 100 mm in diameter, The carbon charge (30 g) is first conditioned at 1000 rpm for 5 minutes, screened at 1.00 mm and dried. The dry oversize (20 g) is returned to the baffled vessel and attritioned for a further 20 minutes. This procedure can be continued if necessary. Attrition resistance is calculated using Equation (3). Wet attrition tests using only water show an inverse relationship between the attrition and the particle size (Table 3).

Table 3, Wet attrition resistances for various activated carbons.

Sample	Particle size (mm)	Wet attrition resistance (percent)
A) Coconut shell	2.80-3.36	91.0
A) Coconut shell	2.00-2.36	92.5
A) Coconut shell	1.40-1.70	93.0
B) Coconut shell	2.00-2.36	93.0
C) Coconut shell	2.00-2.36	88.5
D) Extruded	+1.00 mm	91.5
E) Extruded	+1.00 mm	95.5

Some preliminary data are available for pulp attrition tests on a limited selection of carbons. Figure 2 shows the cumulative loss of carbon (-1.00 mm material) as the test is repeated several times on the oversize remaining from the previous test. Quite significant differences in behaviour were recorded for some commonly available activated carbons.

Wear cycle times for both the wet and dry tests were chosen to give at least a 5% weight loss as fines for the premium commercial carbons.

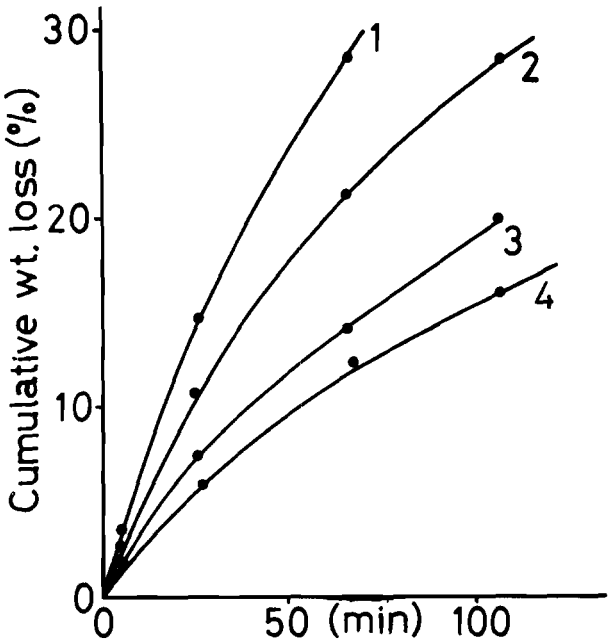

Figure 2. Attrition of carbon in 40% w/v pulp, carbon concentration 10 g/l.

This has made the tests less sensitive to operator and experimental error and gives a greater spread of results allowing for easier comparisons to be made between carbons.

Other Tests

Characterisation of carbons can also involve equilibrium loading tests, Methylene Blue,CTC or Iodine Numbers, bulk density and particle size analysis. Equilibrium loading or capacity is not considered as important as kinetic activity because in practice carbons are never loaded with gold to more than about 10 percent of their capacity. The combination of limited residence time and fouling by other pulp species limits gold loadings to 2000-10,000 g/t. Methylene Blue CTC, or Iodine Numbers are effectively measures of surface area which consequently may reflect activity. However, low activity brought about by fouling does not necessarily result in decreased values of Methylene Blue, CTC or Iodine Numbers. Surface areas based on nitrogen absorption are around 1000 m^2/g for good activated carbons but the way in which the surface is prepared is also important. Bulk density is another useful measure of carbon activity in that it indirectly relates to the extent of activation. The activation process involves treating the carbon with steam at temperatures above 800°C in order to create the pore structure and the right surface chemistry. Development of pores results in loss of carbon and hence lowers the bulk density to around 0.5 kg m^{-3}. Very low bulk densities are associated with very high activity but poor wear characteristics. Finally the particle size range of any batch of activated carbon should average around 2 mm for coconut shell type. Particles larger than 3 mm will have low activity and those below 1 mm will pass through the interstage screens and be lost into the tailings pulp.

It is important to use a combination of tests to properly assess a particular carbon. For example, a carbon submitted for testing showed a high activity, a reasonable dry abrasion resistance but a very low Carbon Tetrachloride Number. Examination of the wear test data revealed a very high pre-abrasion value. Discussion with the client confirmed that the steam activation had been carried out rapidly resulting in over-activation of the outer surface of the coconut shell char. This outer surface was of low bulk density and, consequently, was easily abraded exposing the harder core, which was largely unactivated.

PLANT MONITORING

The activated carbon used for gold recovery in a CIP plant is also an efficient adsorber for many other pulp constituents both organic and inorganic (Labrooy and Bax, 1985). Consequently the activity of carbon decreases with increased contact times in the pulp. The loaded carbon is then separated from the pulp, acid washed, eluted, regenerated and returned to the adsorption section. An activity profile of carbon through a plant is often a useful guide to overall plant performance when compared to the gold loading profile. Table 4 compares the

Table 4, Activity and gold loading profiles of CIP plants treating oxide ores.

	Plant A		Plant B	
Carbon sample	Activity (%)	[Au] (g/t)	Activity (%)	[Au] (g/t)
Fresh	100	-	100	-
Kiln Regen.	55	-	70	-
Adsorber 6	47	80	22	80
5	42	175	15	90
4	36	520	13	270
3	33	1500	12	535
2	33	2140	6	1460
1	33	4500	8	2330
Eluted	40	14	40	50

behaviour of two plants treating oxide ore. Points to note are:

a) Better kiln regeneration in Plant B.

b) Maintenance of higher carbon activity through the adsorption section of Plant A.

c) Lower gold loadings on barren carbon from Plant A.

d) Different gold loading profiles. Plant B has some reserve capacity in the last adsorber (No. 6) to cope with any variations in gold grade or temporary shut down of the regeneration kiln.

e) Significant improvement in carbon activity following elution in the case of Plant B. This particular operation treats lateritic ore from a near surface deposit and the presence of naturally occurring vegetation

products results in organic fouling of the carbon. These organics are probably removed by the strongly alkaline, hot strip solution.

It is often instructive to measure the levels of other species adsorbed onto carbon. Those most often present on carbons from Western Australian CIP plants include inorganics such as silver, nickel, copper, sulphate, carbonate, silica, iron, magnesium and calcium, and organics such as flotation reagents, humic acids and lubricating oils. Detection of organics by thermal analysis methods has proven to be quite successful, particularly for flotation reagents. A common contaminant on activated carbons is calcium carbonate and at levels above 5% w/w there is a profound deactivating effect. Acid washing of such carbons usually results in significant increases in activity.

CONCLUSIONS

The availability of appropriate procedures for measuring the properties of activated carbon is of great value to the metallurgist in making the initial selection of the most cost effective carbon and in the subsequent monitoring of the performance of the carbon in the plant. Those properties of carbon which are of immediate concern appear to be activity, wear resistance and particle size. An effective and simple activity test enables the plant operator to carry out measurements on-site which allows daily checking of the extent of carbon fouling and the effectiveness of the regeneration kiln. Although carbon losses by attrition can only be accurately measured through actual plant operating experience the availability of laboratory test methods is useful for ranking carbons in order of wear resistance. The final decision in carbon selection will also depend on other factors such as availability, consistency of product, moisture content, particle size distribution and last, but not least, price.

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