Microwave heating principles and the application to the regeneration of granular activated carbon

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Synopsis

The principles of microwave heating are discussed with reference to dielectric properties, heating phenomena and microwave equipment. Microwave regeneration of granular activated carbon for the carbon-in-pulp process is examined on a laboratory scale, with special attention being given to highlight the problem areas and specific considerations related to microwave heating. Results indicate that microwave regeneration of carbon with 40% moisture on a wet basis by heating to 650°C with steam addition regenerates the carbon to virgin carbon activity levels but with a higher abrasion resistance. A preliminary economic assessment showed that the capital cost for 120 kg/h microwave unit would be R540 000 with an annual operating cost of R 320/t carbon. These figures yield a return on investment of 12%. The aim of this paper is to provide an introduction to microwave heating and to illustrate its application to the microwave regeneration of granular activated carbon for the carbon-in-pulp (CIP) process.

Microwave heating

Microwave heating, which uses electromagnetic energy in the frequency range 300-3000 MHz, can be used successfully to heat many dielectric materials. Microwave heating is usually applied at the most popular of the frequencies allowed for ISM (industrial, scientific and medical) applications, namely 915 (896 in the UK) and 2450 MHz. Domestic microwave ovens are a familiar example operating at 2450 MHz. The way in which a material will be heated by microwaves depends on its shape, size, dielectric constant and the nature of the microwave equipment used. In the microwave S-band range (2450 MHz), the dominant mechanism for dielectric heating is dipolar loss, also known as the re-orientation loss mechanism. When a material containing permanent dipoles is subject to a varying electromagnetic field, the dipoles are unable to follow the rapid reversals in the field. As a result of this phase lag, power is dissipated in the material. The heating of solid dielectrics can also be explained by modifications of this classical Debye theory (Metaxas and Binner, 1990, Kenkre, 1991 and Katz *et al.*, 1991).

In order to account for the various heating mechanisms (termed loss mechanisms), the dielectric constant for a real dielectric attains a complex form

$$\varepsilon * = \varepsilon' \pm j\varepsilon_e''. \qquad [1]$$

The imaginary term ε_e'' is termed the effective loss factor, and accounts for dipolar relaxation loss as well as any conduction and Maxwell-Wagner losses (generally important only at lower frequencies). In general, ε_e'' is a function of temperature, moisture content, density and electric field direction. Much of the original data on ε_e'' were amassed by Von Hippel, 1954, who presented data for both organic and inorganic materials over a range of frequencies and usually for room temperature. More recently, techniques have allowed measurements to be extended to high temperatures (see e.g. Tinga, 1992 , Arai *et al.*, 1992, Arai *et al.*, 1993). The measurement of dielectric properties remains an area of extensive research.

The effective loss factor controls the power that can be dissipated in a material. If $\varepsilon_e'' < 10^{-2}$ the material is said to be of low loss type, and couples poorly with microwaves. In order for such a material to be heated with microwaves, a very high electric field would be needed and application of the field would be done in a single mode cavity (see below). Such materials would be poor candidates for microwave heating applications. For materials with $\varepsilon_e'' > 5$, the power penetration depth could be quite small. If the object to be heated is larger than this depth it is likely that highly non-uniform heating will result.

An estimate of the volumetric power dissipation can be obtained from the following expression

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$$P_{av} = \frac{1}{2}\omega\varepsilon_o\varepsilon_e^{''}\int(E^*\cdot E)dV \qquad [2]$$

where P_{av} is the average power, ω the frequency of radiation, ε_o the permittivity of free space, E the electric field strength and E^* the conjugate of the electric field strength. This equation shows that greater electric field strengths are required at lower frequencies to obtain the same power density, i.e. a greater electric field would be required at 915 MHz than at 2450 MHz to produce the same power density. Tractable forms of Equation [2] can be used to analyse idealised geometries; application to realistic situations is a demanding specialist activity.

It is also of interest to calculate the power penetration depth D_p, which is defined as the depth at which the power drops to 1/e of its value at the surface:

$$D_p = \frac{\lambda}{2\pi\sqrt{2\varepsilon'}}\left[\left(1+\left(\varepsilon_e^{''}/\varepsilon'\right)^2\right)^{\frac{1}{2}}-1\right]^{-\frac{1}{2}} \qquad [3]$$

where λ is the free space wavelength of incident radiation. This shows that for radio frequency (RF) heating the penetration depth is great, but for the common ISM microwave frequencies 915 and 2450 MHz, D_p may be much smaller than the object size, and may be limited to a thin skin. This can lead to highly non-uniform heating. The treatment for heterogeneous materials is more complex, in which an integral form for the density function is required to calculate effective loss factors (Roussy and Thiebaut, 1994). Standish *et al.*, 1991 have also considered the particle size effect in microwave heating of granular materials.

Many heating applications require a uniform temperature in the object being heated. Achievement of this using microwave heating can be a difficult task, and represents perhaps the major challenge of microwave heating (Sutton, 1992). An understanding of why microwave heating usually results in non-uniform heating, and the nature of the non-uniformity, can be gained by consideration of the heating mechanism. When a dielectric is placed in a microwave field, energy is dissipated in the material; usually the power decays with increasing depth of penetration into the dielectric. Without surrounding insulation material, heat will be lost from the surface of the object, by convection and radiation. Through conduction in the object, heat will be transferred towards the centre of the object. The net result of these two effects is a temperature profile that varies with time. Shortly after commencement of radiation, the maximum temperature is near the surface. As time increases, the position of maximum temperature moves into the interior of the object. The profile that is established will depend on the power, the electric field, the dielectric and thermal properties of the body, etc. Many theoretical and experimental investigations have verified this (see, e.g. Thomas *et al.*, 1994, Binner and Cross, 1993). The inherently non-uniform electric field commonly found in multimode applicators (see below) is also a cause of non-uniform heating.

Microwave equipment

Microwave equipment consists of three major components, the power supply and microwave generator, the applicator, and the control circuitry. The power supply and microwave generator provide microwaves at the appropriate frequency. The power supply must be matched to the microwave source to ensure correct operation. The most common microwave sources are the magnetron and klystron, the former being robust, efficient, frequency stable and readily available while the latter is more expensive, available in higher powers, but with a somewhat longer operating life (80000 versus 10000 hours). Low power magnetrons for laboratory use typically come in the power range 1-6 kW and may be of fixed or variable power type, while higher power magnetron systems up 70 kW are available, usually at 915 MHz rather than the more common low power frequency of 2450 MHz. Power output from low power permanent magnet magnetrons can be achieved by thyristor control of the anode voltage, while high power systems usually use an electromagnet to vary the anode current. It is usual to insert a circulator between the magnetron and the load. This is a 3-port structure that couples power clockwise between adjacent ports. As microwave power can be reflected, the circulator protects the magnetron from excessive reflected power by diverting reflected power to a water-cooled matched load, which may also be equipped with a power meter. The circulator is particularly important in preliminary investigations where the dielectric properties of the load are not well known and where a general purpose applicator is being used which may result in significant amounts of reflected power. To provide a good impedance match between the magnetron and the load a stub tuner is often provided. Even with the use of a circulator it is sometimes found that significant power pulling (20% of nominal power) due to impedance mismatches can still occur (see below). Both the stub tuner and the circulator are expensive and cope with powers of up to about 6 kW. For these reasons they are not generally used in high power industrial systems.

Microwave applicators are metallic enclosures that contain the material to be heated, and their design depends on the processing requirements. Travelling wave applicators are used for processing thin webs of material, and would be of limited use in minerals processing. Single mode cavities, in which the electromagnetic field exists in a well-defined form, are useful for processing small quantities of material (e.g. filaments), particularly those with low effective loss factors. Full descriptions are given in Metaxas and Meredith, 1983. The most common applicator is the multimode cavity, which is basically a large box, with at least one dimension somewhat larger than the free space wavelength of the radiation (122 mm at 2450 MHz). Microwave radiation entering a multimode cavity undergoes multiple reflections to form a complex standing wave pattern, governed by the dimensions of the cavity and the nature of the load. The multimode cavity is versatile and suited to heating large loads and can be adapted for continuous processing. Unfortunately, its convenience is offset by problems of poor electromagnetic uniformity and difficulties in modelling and design. Specialized features such as mode stirrers and slotted waveguide feeds can overcome the former (Kashyap and Wyslouzil, 1977). The mode stirrer is the most common of these devices, consisting of a rotating vaned metallic fan. Rotation speeds are typically 1-10 rev/s (Metaxas and Meredith, 1983). The domestic microwave oven is an example of the multimode cavity, and is often used as a general-purpose cavity for initial laboratory scale investigations. However, domestic ovens are not designed for high temperature operation, have no provision for temperature control and generally have extremely rudimentary power control (by variation of the duty cycle). Although they are useful for initial screening, unless used with care, seriously misleading results can result from such experimentation. Generally, the performance of a multimode cavity will improve as the flling factor increases. Conversely, a small load in a large cavity will result in a large Q-factor meaning that only a small fraction of the energy applied to the cavity is dissipated in the load.

Usual materials of construction for microwave applicators are stainless steel or aluminium. The latter is a good compromise between cost and high electrical conductivity. This is desirable to reduce loss of power through currents set up in the applicator walls and is likely to be important when small loads are heated in large cavities. Doors can be made with 1/4 wave chokes to contain microwave radiation, or more simply by ensuring good electrical contact around the entire sealing interface. This can easily be achieved by using copper braid as a gasket. Feed ports can be provided into the microwave cavity; generally holes of less than 10 mm diameter will act as chokes at 2.45GHz and prevent leakage of radiation. Larger apertures can be provided but require electromagnetic design and possibly need to be provided with absorbing materials and water-cooling. Details on choking can be found in Metaxas and Meredith, 1983.

Because materials heated in a microwave oven lose heat from the surface to the relatively cool interior of the oven, careful attention has to be given to insulation of the workpiece, especially for high temperature applications. In general the insulation material must be microwave transparent at the operating temperature while possessing a sufficiently low thermal conductivity. High alumina, zirconia and silica-alumina refractories are commonly used. If the insulation couples appreciably with microwaves, microwave power will be attenuated and efficiency will be reduced. The alternative is to place the insulation outside the cavity, but if the cavity is large relative to the size of load, this will also be thermally inefficient. Hybrid insulation systems using materials which couple with microwaves at low temperatures (susceptors) have been developed to insulate materials that are difficult to heat with microwaves at low temperatures (Janney *et al.*, 1991). An alternative to the use of susceptors, and one that is much easier to control, is the use of an additional heat source, such as gas firing or resistive heating, to provide an increased ambient temperature. This approach has the added benefit that it is usually much more efficient than using either microwave or conventional heating alone.

The various microwave components are connected by waveguides (generally rectangular section brass) of appropriate dimensions for the operating frequency (86.36 x 43.18 mm at 2450 MHz). A stub tuner is often placed between the load and the magnetron to allow impedance matching. This device can readily be adjusted by hand to minimise reflected power, while automatic tuning is also possible, using stepper motors. A dual directional coupler allows measurement of forward and reflected power. The directional coupler is a passive microwave device that couples a fraction of the power transmitted in a given direction to a detector port equipped with a crystal diode detector. Nominal couplings of 30 and 60 dB are common. In the latter case, the ratio of measured to actual power is 10^{-6} (for a forward power of 1 kW, 1 mW would be measured at the detection port). Calibration of these devices requires considerable expertise and sophisticated equipment. Control circuitry will usually allow temperature control by power manipulation, and sometimes automatic impedance matching.

Microwave safety

Although microwave radiation is a non-ionising radiation it nevertheless poses a safety hazard. It is vital to ensure integrity of all microwave joints and doors. Microwave leakage detectors are readily available for this purpose. A radiation flux of 5-10 mW/cm^2 at a distance 50 mm from the equipment is a generally accepted limit. Doors should preferably be fitted with interlocks and water flow monitors should also be installed. When choking systems requiring resistive choking are used it is essential that safety cut-outs are provided. Compatibility with other communications systems is important, particularly as cellular telephones operate close to the 915 MHz band.

Applications of microwave heating in minerals processing

Microwave heating has been applied to a number of minerals processing applications. Walkiewicz *et al.*, 1988 present data on microwave heating of a number of minerals, and speculate on the potential reduction in grinding energy required for minerals with stress fractures induced by microwave heating. Bradhurst and Worner, 1990 discuss applications of microwave heating in mineral processing and pyrometallurgy, including drying of coal, gold extraction and ore reduction (see also Standish and Huang, 1991 and Standish and Worner, 1991). Haque, 1987 has discussed microwave pre-treatment of refractory gold ores. Rowson and Rice, 1990 examined microwave-enhanced desulphurisation of coal mixed with caustic solutions. The rapid microwave pyrolysis of coal was studied by Monsef-Mirzai *et al.*, 1995. Microwave research areas associated with mineral processing include waste immobilisation using microwave vitrification, and microwave combustion synthesis of ceramics. One of the most promising mineral processing applications, which has reached pilot plant scale, is the regeneration of granular activated carbon.

Microwave regeneration of granular activated carbon for the CIP process

The carbon-in-pulp process is widely used for the recovery of gold. The carbon, which is used to adsorb the gold cyanide molecule, is periodically removed from the adsorption tanks to allow removal of the gold by elution. The carbon is then usually acid washed to remove inorganic compounds and regenerated at 650-850°C in a steam atmosphere to remove other foulants such as flotation reagents, lubricating oils and humic acids which would foul the carbon and reduce its performance. Regenerated carbon is sized and returned to the CIP circuit. Regeneration is conventionally done in rotary kilns or vertical tube furnaces. These may be either electrically-heated or gas-fired. Both units rely on indirect heating of the carbon charge. Direct resistive heating is also used for carbon regeneration, the so-called Minfurn being a continuous version of the earlier Rintoul furnace.

Advantages of the technology are primarily cited to be low maintenance costs. Recently a resistively-heated rotary kiln has been developed.

Microwaves can readily heat granular activated carbon directly, suggesting that microwave regeneration would offer possible advantages over conventional regeneration. These include rapid and precise temperature control of the carbon inventory itself, a more compact furnace and possible energy savings.

Tests on microwave regeneration have been promising. Strack *et al.*, 1995 have developed a pilot plant regenerating 12 kg/h activated carbon by microwave heating at Barrick's Holt-McDermott gold mine in Ontario, Canada. The system used a hot air pre-dryer, followed by a vertical tubular microwave regeneration unit. The activity of the microwave-regenerated material was comparable with that from conventional regeneration, while losses due to attrition and consumption in the kiln were reduced by half. The predicted operating costs for a 120 kg/h unit were 1/3 of a conventional system. A payback time of 15 months was predicted in spite of increased capital costs for the microwave equipment.

The remainder of this paper reports a laboratory scale investigation of microwave regeneration of granular activated carbon that aimed to find a suitable compromise between carbon performance and energy consumption. The processing problems associated with use of microwave heating are highlighted.

Experimental methods

Materials

Spent granular activated carbon from Vaal Reefs no. 8 Gold Plant containing 40% moisture (wet basis) was used in the experiments. This carbon was a mixture of ANK 11, GRC 22 and 207C. Samples of the virgin carbons and mine-regenerated carbon were also tested. For the different adsorption experiments, potassium aurocyanide (as a pure crystalline salt) was used. Glass distilled deionized water was used in the experiments.

Microwave equipment

The regeneration experiments were carried out in a system consisting of a SAIREM GMP12T variable power 1.2 kW, 2450 MHz magnetron, with a 3 port circulator and stub tuner. A 510 x 450 x 440 mm aluminium cavity was used (Chow Ting Chan and Reader, 1996). A second series of experiments, designed to examine the effect of moisture content on carbon properties and energy consumption, was performed using a fixed power 1.25 kW system, equipped with a circulator, stub tuner and 60 dB dual directional coupler. In addition to the cavity described above, a mild steel modified domestic cavity applicator was also used for some experiments. A four-bladed mode stirrer was used to improve electromagnetic field uniformity in the cavity.

150g batches of carbon were contained in a quartz reactor, 290 mm high and with an outside diameter of 45 mm. The reactor was provided with gas inlet and outlet, and ports for the insertion thermocouples. Quartz was chosen as the material of construction as it can withstand high temperatures and is transparent to microwaves. Unfortunately it is brittle and crystallises with extended use.

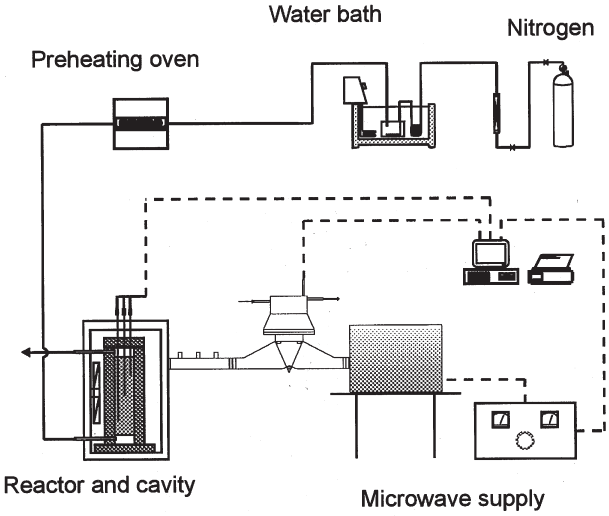


Figure 1—Experimental equipment

The reactor had a preheating section (40 mm deep) below the porous quartz disc at the bottom of the reactor, which could be filled with 30g of virgin carbon. This served to preheat gas entering the bottom of the reactor in an attempt to ensure temperature uniformity through the carbon bed. The lid contained the gas outlet and the three ports for the thermocouples. The thermocouples were inserted directly into the carbon bed. Tapered graphite plugs were used in the ports to prevent any leakage of gas. Asbestos string was used as the packing material between the thermocouples and the graphite plugs. The reactor was insulated in a 72% alumina–28% silica cylinder (Detrick Board 1600DH). Additional insulation between the cylinder and reactor was provided by 40% alumina–48% silica–10% zirconia fibre blanket (Fiberfrax® Durablanket). An additional wrapping of Fiberfrax to a total insulation thickness of 75 mm was provided. Thermocouples were inserted into the carbon bed to depths of 25, 65 and 105 mm. These depths represent the midpoints in the upper, middle and lower zones of the carbon bed. Figure 1 shows the equipment schematically.

Nitrogen saturated with water vapour at 90°C was used to provide an oxidising atmosphere for the oxidation of the pyrolysed adsorbents for some of the experiments.

Temperature and power measurements

Calibration of the forward and reflected power meters on the SAIREM GMP 12T by testing against various water loads was necessary. Measurements using an empty reactor indicated that about 400 W was absorbed in the reactor insulation. Power measurement for the 1.25 kW fixed power system was by means of the 60 dB dual directional coupler equipped with crystal diode detectors. Calibration of the coupler at low power using a network analyser indicated that the couplings were in fact 58.931 dB and 59.877 dB at the centre frequency of 2.45GHz (note that an error of ±3 dB gives an error of about 50% in actual power). Unfortunately, precise calibration at high power is not possible, which is a problem as the entire microwave circuit behaves differently at high powers under loaded conditions. This suggests that if energy consumption is an important parameter in assessing the merits of microwave heating, then the calibration should be done very carefully and considerable care should be exercised in interpreting power measurements. The diode detectors were calibrated against a signal generator with known power output. Inserting a shorting plate immediately after the directional coupler made a simple check of the directional coupler at high power. In theory, forward and reflected power should be the same in this case but this was found not to be so (1486 W forward, 1389 W reflected). It was also found that the results differed depending on the orientation of the coupler (1406 W forward, 1126 W reflected with the coupler reversed). This indicates that there are high power effects in the presence of all the components that cannot be determined from low power calibration

Temperature was measured using 3 mm O.D. type K stainless steel sheathed thermocouples calibrated against the triple and boiling points of water. It was found that 1.5 mm diameter thermocouples self-heated in the electromagnetic field. Olmstead and Brodwin, 1997 have developed an analysis showing that the temperature measured by the thermocouple is strongly dependent on sheath thickness. Thermocouples must be of the ungrounded tip variety, while grounding of the metallic sheath to the cavity is required to avoid arcing. Shielding the thermocouple circuit from the high voltage present in the microwave circuit required considerable effort. It was possible to reduce interference to within the thermocouple resolution (± 1.5°C). General guidelines in this regard are to use shielded extension cables which should be earthed to the thermocouple amplifier circuitry and to remove such circuitry as far as possible from the high voltage area. The amplifier circuitry also needs to be designed to exclude electromagnetic interference from the high voltage circuit. Temperature control was based on the average bed temperature. Temperatures were sampled every 0.5 s. Grellinger and Janney, 1993 provide a comprehensive discussion of the three most commonly used temperature measurement techniques in microwave heating *viz*., thermocouples, infra red (IR) pyrometry and optical fibre thermometry (OFT). They found agreement within ±20°C for the 3 types of measurement when tested for microwave heating of zirconia and alumina. IR and OFT have the drawback that they measure surface temperatures, although neither are affected by electromagnetic interference. Optical fibre probes can also be used as contact sensors but are not robust enough for industrial use. They are also extremely expensive.

Regeneration experiments

Carbon was regenerated in the temperature range 650–750°C. Prior to regeneration the reactor was purged with nitrogen gas to create an inert atmosphere. Steam was added to the reactor at the rate of 0.18 kg/h in nitrogen (55 mol% in the feed stream). This is a relatively high steam rate, and corresponded to steam addition ratios ranging from 0.34-0.78 kg steam/kg carbon, depending on the regeneration time. This compares with 0.03-0.17 kg steam/kg carbon for tests on a Rintoul (Cole *et al*., 1986), 0-1.3 kg/kg for tests on a Minfurn (Van Staden and Laxen, 1991) and the recommendation of 0.5-1 kg/kg (Urbanic *et al*., 1985). Van Vliet showed that the rate of oxidation of a typical adsorbate was insensitive to steam additions above 25 mol% for dry carbon, while Van der Westhuysen, 1992 predicted that for carbon containing more than 10% moisture additional steam should be unnecessary. Commercial regeneration is often done without steam addition; moisture evaporated from the carbon provides the oxidising atmosphere. The carbon was heated to the desired regeneration temperature, as indicated by the average temperature, and maintained at that temperature for 0, 5, 10 or 15 minutes. The carbon was cooled under nitrogen and then air prior to analysis. A further set of experiments using no steam or gas flow was conducted in the fixed power system. Carbon of moisture contents ranging from 0–40% on a wet basis was heated to 650°C. This carbon has not yet been analysed (see below).

Carbon testing

The microwave regenerated carbon was assessed using industry standard tests (Van Wyk, 1997) for activity, loading capacity, and abrasion resistance factor. The activity, defined as the dimensionless gold concentration (the gold concentration at time *t* scaled with the initial concentration) after 60 minutes of adsorption in a 20 ppm gold solution, indicates the rate of gold adsorption. The loading capacity is a measure of the carbon's ability to remove gold in the final stages of the CIP circuit. The abrasion resistance factor is a measure of the carbon's ability to withstand attrition in the CIP circuit. In general there is a trade-off between activity and abrasion resistance factor. McArthur *et al*., 1987 have shown that some highly active carbons actually perform poorly after attrition as they lose the soft but active outer layer during attrition. This is detrimental in terms of increased barren losses as well as gold losses with the abraded fines. This suggests that carbon activity should preferably be assessed after regeneration and that a regeneration scheme that can produce a more uniformly hard yet active carbon would be desirable. The recommended performance levels are a loading capacity > 45 kg Au/ton of carbon (approximately equivalent to an equilibrium K value of 26 kg/t), a dimensionless activity > 0.7 and an abrasion resistance factor > 75%. Details may be found in Van Wyk, 1997. Although none of these tests can be used to predict plant performance directly, they are useful in assessing the degree of regeneration and in comparing different regeneration schemes. The results were compared with those for virgin carbon, as well as with carbon regenerated in a 12 tpd rotary kiln and in a 3 kg/h Minfurn. The reproducibility of the sampling procedure has been discussed by Van Wyk, 1997.

Results and discussion

Carbon performance

The results of the activity, loading capacity and abrasion resistance factor tests, together with comparisons with mine-regenerated material, virgin carbon and Minfurn-regenerated carbon are given in Table I. It can be seen from the data that the activity of the microwave-regenerated material is close to that of virgin carbon (which was slightly worse than the recommended performance level). The loading capacity generally exceeded that of virgin carbon, while the abrasion resistance factor was much higher than the recommended performance level. Microwave regenerated carbon generally

Table I

Results of microwave regeneration of activated carbon. Regeneration conditions give highest average temperature reached, time at that temperature and steam addition in kg steam/kg carbon

Regeneration conditions	Activity (–)	Loading capacity (kg au/t carbon)	Abrasion resistance factor (%)	Specific energy consumption (kWh/kg dry carbon)	Average standard deviation of bed temperature (°C)
Virgin carbon	0.67 (100%)	40.5 (100%)	79	–	–
Mine-regenerated	0.51 (76%)	39.5 (98%)	88	1.5-2.3	–
Minfurn	0.67 (100%)	45.0 (111%)	93	1.2	–
Microwaves					
650°C, 0 mins 0.34 kg/kg	0.65 (96%)	44.7 (110%)	96	1.47	112
650°C, 5 mins 0.52 kg/kg	0.62 (93%)	45.7 (113%)	92	1.89	80
650°C, 10 mins 0.62 kg/kg	0.60 (89%)	45.3 (112%)	92	1.91	112
650°C, 15 mins 0.72 kg/kg	0.58 (87%)	44.5 (110%)	91	2.02	154
700°C, 0 mins 0.41 kg/kg	0.60 (89%)	45.8 (113%)	92	1.74	151
700°C, 5 mins 0.52 kg/kg	0.63 (94%)	46.1 (114%)	92	1.91	48
700°C, 10 mins 0.59 kg/kg	0.65 (96%)	46.3 (114%)	92	1.87	69
700°C, 15 mins 0.77 kg/kg	0.68 (101%)	47.2 (117%)	93	2.32	106
750°C, 0 mins 0.40 kg/kg	0.68 (101%)	46.6 (115%)	93	1.73	100
750°C, 5 mins 0.48 kg/kg	0.66 (99%)	47.3 (117%)	92	1.81	142
750°C, 10 mins 0.59 kg/kg	0.71 (105%)	47.8 (118%)	92	1.88	75
750°C, 15 mins 0.78 kg/kg	0.72 (107%)	47.6 (117%)	92	2.30	104

performs better than mine-regenerated material, while the carbon regenerated in the Minfurn performed as well as the best of the microwave regenerated carbon in terms of activity and loading capacity. Statistical analysis of the results showed that at a significance level of 0.05, different regeneration times did not affect any of the carbon properties, while different regeneration temperatures affected only activity and loading capacity. This finding confirms conclusions drawn by *inter alia* Van Deventer and Camby, 1988, Cole *et al.*, 1986, Van Vliet, 1991 and Avraamides and La Brooy, 1988.

A number of other studies confirm that regeneration for short times at 700–750°C provides good carbon properties. Strack *et al.*, 1995 found that carbon activity could be restored to 105% of the virgin carbon value by heating with microwaves to 700°C. No steam was added to carbon with moisture content of 8%. Avraamides *et al.* found that adequate regeneration could be achieved at temperatures less than 750°C with a total steam addition of 0.2 kg/kg.

It is difficult to specify an optimum set of regeneration conditions, as there is a trade-off between activity and abrasion resistance factor. Following the approach of McArthur *et al.* one could define an optimum set of conditions as those producing a carbon with as high an activity as possible after attrition, while retaining sufficient hardness to be acceptable for plant operation. It seems unlikely that a relationship between an abrasion resistance factor tested in the laboratory and abrasion losses on a plant will be developed in the foreseeable future. From the present results it appears that microwave regeneration at 650°C for 0 minutes is satisfactory; the activity is close to that of virgin carbon, while the abrasion resistance factor is also very high. A carbon of high activity will reduce the barren solution value, decrease gold lock up on the carbon inventory and thereby decrease the gold loss due to carbon breakage (Bailey, 1987).

Strack *et al.*, 1995 found that losses due to attrition were reduced and the hardness of the material improved for microwave regeneration. Microwave regeneration was found to reduce by half the loss of carbon; a saving of 40 kg per ton of carbon processed was predicted. This is significant in terms of gold and carbon savings, as fines abraded from the carbon carry with them valuable gold. There thus exists potential for considerable costs savings.

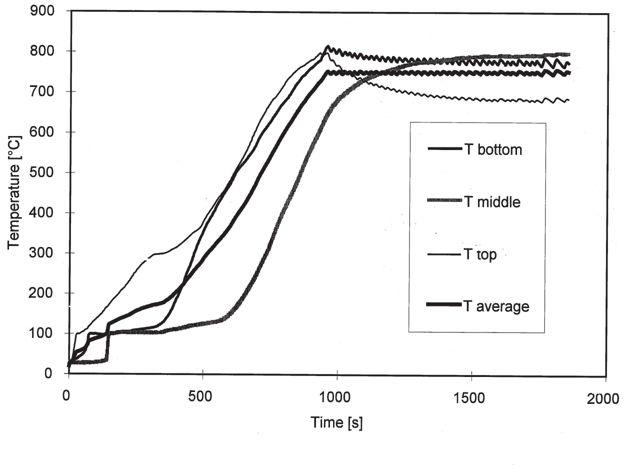
Steam addition

There is evidence that steam addition is not necessary if the carbon moisture content is more than 10% (Van der

Westhuysen, 1992, Cole *et al.*, 1986, Van Staden and Laxen, 1991). This result is borne out by current industrial practice. In the present study, steam to carbon ratios ranged from 0.34-0.78 kg/kg, which is much more than necessary. Further work is underway to determine the optimum moisture content for regeneration without the need for supplementary steam. The use of supplementary steam on a plant would require a package boiler, and would increase the heat load on the regeneration system due to superheating requirements.

Temperature uniformity

Figure 2 shows the temperature profile for regeneration at 750°C for 15 minutes. A rapid initial heat up to 100°C is followed by a period of moisture removal in the middle portion of the carbon bed, while the top dries out rapidly. In general it was found that experiments performed with gas flow and steam addition showed a maximum temperature at the top of the bed while for those done without gas flow, the lower part of the bed was hottest and the centre coolest. The former effect may be due to the cooling effect of incoming gas. Modelling efforts presently underway should elucidate these phenomena. The profiles shown in Figure 2 are typical in that they show considerable temperature variations across the bed depth. It is likely that the main cause of temperature non-uniformity is differential drying. Tests on pre-dried carbon show better uniformity, as would be expected. Other reasons for lack of temperature uniformity are inherent electromagnetic field non-uniformity, non-uniform heat loss and sample inhomogeneity. These effects are difficult to predict *a priori* most especially because modelling the electromagnetic field distribution in the cavity and load, and coupling this to the temperature and moisture profiles, is an extremely demanding task. (Constant *et al.*, 1996 have modelled microwave drying, and while the heat mass transfer phenomena were modelled comprehensively, the electromagnetic field distribution assumed exposure of the load to a plane wave, which is a major simplification, and would be inappropriate in the present study.) Examination of the temperature profiles showed that the degree of temperature uniformity differed from experiment to experiment. Reasons for this were not immediately apparent, but are probably due to variations in position of the reactor in the cavity, in which the electromagnetic field is inherently non-uniform. Unfortunately, spatial temperature gradients are likely to result in non-uniform regeneration. The sampling procedure used in this investigation thoroughly mixed the carbon prior to analysis, and repeated tests showed that analysis was reproducible (Van Wyk, 1997). The average standard deviation in bed temperature in °C for each regeneration experiment is shown in Table I.



Temperature profile during microwave regeneration at 750°C for 15 minutes

There are a number of other measures which would increase temperature uniformity, including pre-drying, using a cavity with a greater filling factor (to decrease the Q-factor) and continuous processing. Using mode stirrers, slotted waveguide feeds, multiple feeds and complex insulation systems are all appropriate methods for increasing electromagnetic field uniformity, and hence temperature uniformity. The best approach would be continuous processing, which would probably render these methods unnecessary, and would be operationally more convenient in a plant situation. Temperature uniformity may be affected by penetration depth. Operation at the more common high power frequency of 915 MHz would help, as the penetration depth increases with decreasing frequency.

Energy consumption

Accurate prediction of process energy consumption from laboratory scale microwave testing is difficult. Reasons for this include: the effect of scale factors (surface to volume ratio and heat loss effects), difficulties in calibration of power sensors especially at high powers and other measurement problems, non-optimal applicator designs with high reflected powers, small filling factors giving large wall losses, especially in stainless steel cavities, and uncertainty in predicting magnetron efficiencies at high powers. Microwave energy consumptions are reported in Table I. Statistical analysis showed that only time had an effect on energy consumption at a significance level of 0.05, and as carbon properties were relatively insensitive to regeneration there would be little point in regenerating for extended periods. Based on a measured electrical conversion efficiency of 70%, the total electrical energy consumption (1.47-2.29 kWh/kg) for microwave regeneration lies in the range typically expected for a rotary kiln (1.5-2.3 kWh/kg dry carbon), and a full scale Minfurn (1.2 kWh/kg). Magnetron efficiency at 915 MHz for a large power unit could be 85%, which would improve the total electrical energy consumption. Penetration depth also increases at 915 MHz, which would allow greater freedom in selecting the dimensions of the applicator. A plug-to-product efficiency of 50-70% on an industrial scale is generally accepted norm (Metaxas, 1991).

Further reductions in energy consumption could be achieved by using no steam, and by optimising the applicator design, rather than using a general-purpose multimode applicator. It is unlikely that an industrial process would use microwave energy alone, and it may be that hot air pre-drying of the carbon followed by further radio frequency or microwave drying, followed by combined microwave and resistive heating would be best. To decide this, it would be necessary to know the optimum moisture content for steam-free regeneration, in terms of carbon properties. Preliminary tests on this have begun, using the fixed power 1.25 kW system. It has been found that the energy consumption for microwave heating to 650°C obeyed the following relationship:

$$E=0.0164M_C+1.480 \qquad [4]$$

where E is the energy consumption in kWh/kg and M_C is the moisture content as a percentage on a wet basis. Measurements indicated that about 400 W were absorbed in the cavity and insulation. It was also found that there was power pulling of about 400 W in the microwave circuit (i.e. the forward power of the magnetron was about 400 W than would be expected from the normal operating characteristics). This is undesirable as it can lead to magnetron operation outside the design range resulting in magnetron damage. The use of the circulator would have been expected to prevent this. The energy consumptions predicted by Equation [4] are greater than those reported in Table I. This could be due to the different microwave sources, the different location of the reactor in the cavity, the difference in power measurement system and the fact that the drying was done without flow of gas. These discrepancies highlight the difficulties in predicting energy consumption from small-scale tests.

In tests on 2.5–6 kg batches of carbon, Haque *et al.*, 1993 found that the average specific energy consumption for microwave regeneration at 700–750°C for 5 minutes at 915 MHz was 2.38 kWh/kg. The moisture content was 50% on a dry basis, and it was estimated that 70% of the energy requirement was for drying. On the 12 kg/h pilot plant, 1 kWh/kg was used for predrying carbon of 40% moisture (wet basis) to 8% in the hot air pre-dryer, and 1.1 kWh/kg was used for microwave regeneration.

Process economics

Van Wyk, 1997 has presented a preliminary estimate of the process economics, based on the energy requirements discussed above and capital and operating cost estimates obtained from industry. As a rule of thumb, microwave equipment will cost about $1200/kW for high power (915 MHz, 60 kW) units. Based on the work of Van Wyk, capital and operating costs for three different regeneration systems processing 120 kg/h are presented in Table II. The return on investment, defined here as the ratio of annual cost savings divided by the capital cost, is 12% for the microwave unit compared with 15% for a Minfurn. This result is sufficiently promising to merit further investigation. If it can be shown that microwave regeneration results in a significantly harder product for the same activity then there could be further economic advantage to the use of microwaves. Carbon losses from CIP plants are in the range 30–40 g/t ore-processed, but can be as low as 2 g/t and as high as 50 g/t. Those plants with high carbon losses would stand to benefit most from a regeneration process that reduces carbon losses. Strack *et al.*, 1995 predicted a payback time of 15 months for a 120 kg/h plant microwave unit.

Table II

Estimated capital and operating costs for different regeneration schemes based on a throughput of 120 kg/h

	Microwave 120 kg/h	Minfurn 125 g/h	Rotary kiln 120 kg/h
Operating cost R/t carbon	320	315	395
Capital cost R	540 000	434 000	428 000
ROI%	12%	15%	–

Microwave benefits

It appears from this work, and that of Strack *et al.*, 1995, that microwave regenerated granular activated carbon has similar activity to that of conventionally regenerated material but has a higher abrasion resistance factor. It is not clear why this should be so, although the effect will be due only to the differing thermal treatment experienced by the carbon. For example, in the Minfurn, most of the current paths lie along the perimeters of the carbon particles and the positions of highest temperature are at particle-particle contact points. There is no evidence in the literature for the existence of microwave effects. All such effects can be explained as thermal effects, although it is certainly true that microwave specific thermal effects can be achieved.

Scale up and technology transfer

While microwave heating is an accepted domestic technology, growth of the industrial heating sector is slow, and most research applications of microwave heating are not commercialised. Indeed, worldwide sales of industrial microwave equipment companies were estimated at only $50 million in 1994 (Krieger, 1994). Obviously much effort is required to ensure commercialisation of a microwave heating technology.

The primary requirements for successful technology transfer are a compelling advantage to the use of microwaves (Stein *et al.*, 1994), and the need for the technology from industry (Tinga and Sutton, 1993, Krieger, 1994). Tinga and Sutton, 1993 have identified a list of obstacles preventing the widespread use of microwave technology; these include the need for custom design for each application, costly equipment and unrealistic expectations. The use of modular equipment design can help to overcome the former, while education will help with the latter. Sutton, 1993 confirms these points.

Stein *et al.*, 1994 attempted to assess the current state of microwave heating technology. They concluded that the successful applications of the technology were those which showed a compelling advantage in the use of microwaves, while those that failed typically did so for rather general reasons such as use of poorly designed equipment for the particular application. This is an important point and should be remembered when using laboratory equipment (which is typically a general-purpose multimode cavity) to assess processes for commercialisation. It was also considered important to take advantage of particular features of microwave heating. The ease of retrofitting microwave technology should also receive strong consideration. Hybrid heating systems were assessed as being very useful in overcoming the problem of temperature uniformity. The importance of dielectric properties and system geometry should also be taken into account.

Krieger, 1994 stresses the importance of taking a systems approach to heating problems, rather than a purely microwave approach. This is very sound, as for many applications it is unlikely that microwave heating alone will be commercially viable (the conversion efficiency of electricity to microwaves is at best 70–80%). Similar conclusions were drawn as long ago as 1987 by Das and Curlee, 1987 who were considering microwave sintering of ceramics. In spite of this there is still a vast amount of research being done on that topic with few, if any, commercial installations. Should a hybrid heating approach be followed, using combined heating for example, it would be desirable to incorporate skills of kiln specialists as well as microwave specialists in the design of the equipment.

Conclusion

Microwave heating is a sophisticated electroheat technology requiring specialist knowledge and expensive equipment if meaningful results are to be obtained. In combination with other heat sources it can offer considerable processing advantages. Due to the heating mechanism, which is entirely different from conventional convection, conduction or radiant heating, design and operation of microwave heating equipment requires solutions to problems that are not amenable to conventional heating heuristics. Microwave heating has been successfully applied to regeneration of granular activated carbon for the CIP process, yielding an active carbon that appears to have a high abrasion resistance factor. Good carbon performance can be obtained by heating to 650°C in a steam atmosphere. Preliminary economic estimates show that there is sufficient merit in pursuing the technology further.

Acknowledgements

ESKOM and MINTEK provided financial support. Vaal Reefs No. 8 Gold Plant supplied the activated carbon. Petrus van Staden of Mintek supplied information on the Minfurn and arranged for use of the 3 kg/h unit. Patrick Chan and Howard Reader of the University of Stellenbosch designed the multimode cavity.

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◆

Wits appointee seeks to balance the scales*

Professor Winston Marasi Onsongo has been appointed director of undergraduate engineering education at the Faculty of Engineering at the University of the Witwatersrand.

One of Professor Onsongo's major goals is to right the imbalances in terms of well-qualified black and female engineers. The best way to create a balance in terms of race and gender is through the introduction of proper support systems for under-prepared students, he believes.

'Students should apply to the Faculty if they want to realize their dreams. We plan to give them the best opportunities and assistance we can, and will admit a few students below our automatic entry level but with potential.'

Professor Onsongo attended the University of Canterbury at Christchurch in New Zealand where he obtained a BE (Hon) in Civil Engineering. He later furthered his studies at the University of Toronto where he obtained MASc and PhD degrees in Structural Engineering.

After working as an engineer he moved to academia and lectured at the University of Nairobi for many years. He was then appointed professor of Civil Engineering at the University of Durban-Westville in May 1994 before joining Wits at the beginning of 1998.

◆

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Dust pollution highlighted at Wits poster day*

Wits University final year chemical engineering student Charles Pool explains his poster on dust pollution to Mr Xavier Prevost of the Minerals Bureau (left) and Wits University's Professor Philip Lloyd at the exhibition held at Wits to mark World Environment Day in June. Environmental engineering, taught by ProfessorLloyd, is part of the final year chemical engineering syllabus at Wits. The exhibition, now in its third year, provides a platform for students and industry to meet and network.◆

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Concern shown for environment at Wits poster day*

Wits University final year chemical engineering student Ronald Mudau explains his poster on disposal of spent catalyst from Sasol to Dr Zoë Butnick-Lees, deputy director-general at Gauteng's Department of the Environment while Wits University's Professor Philip Lloyd looks on. Ronald was one of several exhibitors at the presentation held at Wits to mark World Environment Day in June. Environmental engineering, taught by Professor Lloyd, is part of the final year chemical engineering syllabus at Wits. The exhibition, now in its third year, provides a platform for students and industry to meet and network. ◆

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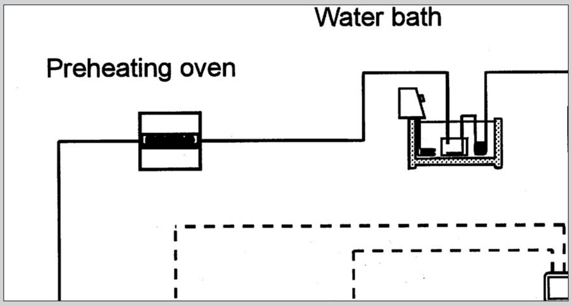
Tuks and Wits Mining Engineers compare strengths on the rugby field*

The 21st annual friendly rugby match between the Mining Engineering departments of the University of Pretoria and the University of the Witwatersrand recently took place in Pretoria. Tuks won by a narrow margin, 15—14. This match is a means of strengthening the ties between these two world-class mining institutions. ◆



The rugby teams of the departments of Mining Engineering of the universities of Pretoria and the Witwatersrand

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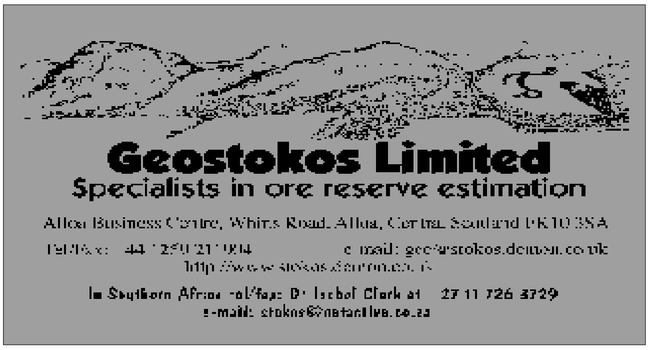

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