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MEASURING FLOC STRUCTURAL CHARACTERISTICS

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Abstract

A review is presented of a range of techniques for the structural characterisation of flocs. Flocs may be considered as highly porous aggregates composed of smaller primary particles. The irregular size and shape of flocs makes them difficult to measure and quantify. A range of different equivalent diameters are often used to define the floc size and allow comparison with other floc systems. The application of a range of floc sizing methods have been described. Microscopy is time consuming, requiring large sample size and considerable preparation but gives good information on floc shape and form. Light scattering and transmitted light techniques have been used to good effect to measure floc size on-line whilst individual particle sensors have limited applicability to measuring floc size. Fractal dimension can be measured using one of three major techniques: light scattering, settling and two dimensional (2D) image analysis. Light scattering is ideally suited for small, open flocs of low refractive index whilst settling may be applied to most floc systems of low porosity. 2D image analysis requires flocs to have good contrast between the solid in the floc and the background.

1. Introduction

The aggregation of fine particles and colloids into larger assemblages is a process that can occur both naturally and artificially. The resulting aggregates that form are known as 'flocs' which are best described as being highly porous, irregularly structured and loosely connected aggregates composed of smaller primary particles (Dolfing, 1987, Huang, 1994 and Kim *et al.*, 2001). In natural aqueous environments examples of floc formation include the transport and deposition of particulate matter in estuaries (Manning and Dyer, 1999), the assemblage of marine particles (e.g. plankton, organic matter, faecal material and minerals) into large aggregates known as marine snow (Ransom *et al.*, 1998) and the colloidal aggregates that are present in most natural surface waters (Gregory, 1997). However, industrial processes that require the separation of solids from liquids may be enhanced by an artificially induced floc formation stage. This includes bioprocess, chemical and mineral processing industries and at water treatment works (WTW) and wastewater treatment works (WWTW) (Zhang *et al*., 1999).

The size and structure of flocs are considered fundamental to the operation of industrial unit processes (Waite, 1999). In water and wastewater treatment processes, the aim is to remove impurities from water in the form of solid particles. Once the solid particles are produced, they may be separated from water using sedimentation, flotation, filtration and thickening techniques (Rebhun and Lurie, 1993). The physical characteristics of the floc are therefore fundamental in determining their removal efficiency. For example, large compact flocs have a high settling rate that results in a treated water of low turbidity during settlement (Wilen *et al.*, 2003), whilst large and porous flocs aid filtration due to high permeability (Bushell *et al*., 2002)

Quantifying floc characteristics is made difficult due to the highly irregular three-dimensional structure of flocs and their inherent delicate nature. In addition, the characteristics of flocculated aggregates have been shown to change depending upon the physical and chemical conditions prevailing in the flocculator (Farrow and Warren, 1989). However, a review of the literature shows there is a comprehensive amount of work on the evaluation of floc structures. The principal aim of this review is to assess how floc structural characteristics can be measured.

2. Coagulation and Flocculation

An in depth review of coagulation and flocculation theory is beyond the scope of this review, for a more rigorous and thorough treatment readers are referred to Gregory (1989) and Amirtharajah and O'Melia (1990). However, in order to understand the importance of floc structure it is firstly necessary to briefly review current knowledge of floc structure and formation. In aqueous systems, floc aggregates are composed of smaller sub-units. In most cases the primary particles are often of sizes between 1 nm and 1 μm consequently fall into the colloidal size range.

There is some confusion in the literature as to the precise definitions of coagulation and flocculation and there appears to be a certain amount of interchanging between the two (Jefferson and Parsons, IN PRESS). However, there is some consensus that the two should be treated as separate and for the purposes of this review the following definitions are used from Cornwell and Bishop (1983) and Gregor *et al*. (1997). **Coagulation** is the process of chemically changing colloids so that they are able to form bigger particles by coming close to one another. This may be achieved by particle destabilisation through double layer compression, enmeshment, chemical

reaction or chemical sorption. **Flocculation** is the process of transferring coagulated colloids into contact with each other to form larger aggregates (flocs).

The exact process of particle destabilisation and the subsequent colloid aggregation is complex. It is generally considered to be a two stage process of particle transport and particle attachment (Thomas *et al*., 1999). The schematic in Figure 1 shows a simplified view of the steps involved. Floc formation is considered a balance between aggregation and breakage (Biggs and Lant, 2000).The rapid initial formation of microflocs is dominated by aggregation, however the importance of floc breakage increases until a steady state floc size is reached.

3. Floc structural properties and their measurement

The evaluation and quantification of floc structural characteristics is made difficult due to the highly irregular three-dimensional structure of flocs and their inherent delicate nature and porosity. However, there is a great deal of information in the literature on methods for the quantification of flocs. The following section deals in turn with measuring floc size, shape and fractal dimension.

3.1 Floc Size

Many different measurements have been chosen as the representative characterisation of floc size. A simple measure of floc size is the floc longest dimension. On its own this measurement is of limited use as it only gives an indication of floc size in one dimension. A more common approach is to find the longest dimension of the floc in both the horizontal (d_{hor}) and vertical (d_{vert}) planes as shown in Figure 2 (Farrow and

Warren, 1989 and Manning and Dyer, 1999). This also allows an indication of the floc height: width ratio and gives an indication of floc shape.

Typically, when referring to floc size a floc equivalent diameter measurement is made (Cousin and Ganczarcyk, 1998). The use of equivalent diameters allows the particle to be defined as a sphere or circle that is in some way equivalent to the particle. Such a standardised measurement allows a comparison to be made between very irregular forms. However, unless the particle being measured is a sphere, then each of these different diameters will take a different value for the same particle. Rather than an absolute value, equivalent diameters should be used for comparative purposes. For this reason it is important that the choice of equivalent diameter remains the same when comparing floc size. Dharmarajah and Cleasby (1986) list fifteen different characteristic diameters that are used to quantify non-spherical particles. Some of these are not applicable to the measurement of flocs because they would damage the fragile aggregates. This precludes the use of sieve diameters, which involves passing the aggregates through a sieve and determining the smallest mesh size that will allow the particle through. The most common size measurements that are used in the literature for flocs are summarised in Table 1.

As microscopy has been widely applied in particle sizing (Allen, 1997 and Aguillar *et al.*, 2003) most of the floc diameters in Table 1 are from two-dimensional images. As with all two dimensional measurements of complex and irregular three dimensional structures, there are difficulties in getting representative size data from a single measurement. Additionally, the results strongly depend upon the orientation of the floc presented to the researcher because a single non-uniform shape has an infinite

number of linear dimensions, so it is only when these results are averaged for a large number of particles that a meaningful number can be found. The British Standard for microscope counting suggests that a minimum of 625 particles should be sized in order to get a representative size distribution (BS3406, 1963).

The diameters based upon 2 dimensional images (such as the projected area diameter (d_a), the Martin's diameter (d_M) and the Feret's diameter (d_f)) are known as statistical diameters because they are only an acceptable indication of particle size distribution if enough measurements are made. The situation is complicated because particles have a tendency to orientate themselves on slides such that they present their maximum area (Allen, 1997). This means that the dimension perpendicular to the viewing plane is generally the smallest and is often neglected. Therefore there is a tendency for statistical diameters based upon 2 dimensional images to be larger than those based upon 3 dimensions. Martin's and Feret's diameters also rely upon the random orientation of the floc in the plane parallel to the viewing direction if only a single measurement per floc is taken from a fixed direction. This confers an advantage on the use of the projected area diameter (d_a). However the advent of image analysis tools has enabled the quick estimation of a range of single measurements to be taken from around the same floc from any number of different directions such that an average value can be used, removing the need for random orientation in this plane. It should also be noted that if the projected area diameter is obtained from a randomly orientated particle in all 3 dimensions (d_p) then the diameter should be representative of the particle in all 3 dimensions as opposed to only 2 and thus provide a more accurate representation of the overall floc size. Indeed, if any of the other statistical

diameters are obtained from the projected area of randomly orientated particles then they too should be representative of the three dimensional floc.

Particle size estimates based upon volume are particularly useful for settlement purposes. The settlement of flocs is a particularly important operational parameter because increased rates of floc settlement results in better solids removal in settlement tanks. In laminar flow, particles fall randomly so orientation should average out over a range of measurements. In non-laminar conditions, particles tend to orientate themselves to resist motion, so the free falling diameter found is smaller than the Stoke's diameter. It is therefore recommended that laminar conditions are applied in order to find a more representative indication of particle size.

3.2 Floc Shape

An indication of floc shape may be provided by sphericity and circularity shape factors. The indices measure how much a particle varies from a sphere or a circle (Equations 1 and 2). The sphericity factor is a function of the volumetric diameter (d_v) and the surface area diameter (d_s).

$$Sphericity = \left(\frac{d_v}{d_s}\right)^2 \qquad \text{Equation 1}$$

$$Circularity = \frac{P^2}{4\pi A} \qquad \text{Equation 2}$$

Circularity is related to the perimeter (P) and the projected area of the particle (A). A value of close to zero indicates a shape approaching a straight line, whilst a value of 1 indicates the shape is a perfect sphere or circle. The shape factors may be of use to

show the change of particle shape under differing conditions. For example, Cousin and Ganczarczyk (1998) compared the affect of salinity on activated sludge flocs. A reduction in the value of the circularity shape factor with increasing salinity indicated that the flocs were becoming more elongated at higher salt concentrations.

4. Methods for determining floc size and shape

Most of the methods for determining floc strength rely upon some measurement of floc size before and after an energy input. Techniques for quantifying and measuring floc size and shape parameters are made difficult due to the inherent irregularity of floc structures in both two and three dimensions. Most efforts have been to size flocs from magnified images captured from cameras (Wang and Gregory, 2002). Two fundamental difficulties arise when using such an approach. The first is which comparable floc characteristic(s) should be measured and the second is how the flocs are prepared prior to being measured.

Farrow and Warren (1993) have divided some of the methods used for characterising aggregate particle size into a number of separate categories (Table 2). Physical sizing techniques such as sieving are inappropriate for aggregates due to their delicate nature. Different workers have used a variety of different methods for the determination of aggregate size. It is important to ensure that the extraction, preparation and measuring technique:

i) measures a representative sample or sub-sample of the original floc suspension

ii) does not damage, break or change the flocs

iii) does not encourage further aggregation

4.1 Microscopy

Microscopy is one of the most widely used technique for measuring particle size (Allen, 1997; Aguillar *et al.*, 2003). Microscopy has been used for decades as a method for sizing and counting flocs (Li and Ganczaryck, 1986; Droppo *et al*., 1996). Before the advent of image analysis gaining useful floc size data was laborious and highly dependent upon the skill of the microscopist. A non-biased selection of flocs is required that is representative of the flocs contained within the sub-sample presented to the researcher. Aggregate size is estimated by reference to a graduated eye piece graticule or by placing flocs in cells with background grids or scales of a known size (for example a plankton counting chamber). This also requires good practice on behalf of the microscopist.

In most instances, carefully dropping a small sample of the suspension onto a microscope slide or into a measuring cell on a slide is suitable for particle size analysis under a microscope (Spicer and Pratsinis, 1996; Wang and Gregory, 2002). In some studies, a cover slip is placed over the sample floc sample as it removes depth of field problems (Cornelissen *et al*., 1997; da Motta *et al*., 2001). However, it is difficult to accept that this approach does not change the flocs due to their delicate nature. The compression from the cover slip is likely to considerably change floc structure.

The problem associated with all techniques where flocs must be removed *ex situ* from the suspension arises from the method of aggregate extraction and preparation prior to being sized. As reported in Farrow and Warren (1993), Camp (1968) used a dipped tube technique, whereby a hollow glass tube was submerged in a flocculated

suspension and sealed at the top and then removed. The tube was then placed horizontally beneath the microscope for analysis. This method provides a good representative sample of the flocs without much rupture. However, in the tube flocs can settle onto one another and cannot be distinguished from one another. Therefore this cannot be a true representation of the actual size distribution. Wang and Gregory (2002) use a similar method to withdraw flocs for analysis using a sampling tube. The contents of the tube were carefully emptied into a microscope cell previously filled with water. By ensuring the tube was large enough and the cell filled with water no floc breakage was seen. In addition, the dilution upon entering the cell should prevent the flocs from falling onto one another. In addition it is important that the technique must not allow further agglomeration to take place during the sample preparation, therefore as dilute suspensions as possible are favoured.

Cousin and Ganczarczyk (1998) and Gorczyca and Ganczarczyk (1999) have used an agar solution (1.5-4 %) to solidify activated sludge flocs and alum flocs in suspension in a Petri dish. A thin layer of the solidified suspension containing an equal distribution of flocs can then be viewed and measured under a microscope. This technique aims to ensure the flocs randomly orientate themselves in the suspension and overcome the non-random orientation of flocs settling onto the slide surface (Farrow and Warren, 1993). Effectively a static measurement is giving a three dimensional representation of the floc. However, it is not clear what affect the agar has floc structure. A further method from Gorczyca and Ganczarczyk (1999) embedded cubes of agar-solidified alum floc suspensions in a hardening resin. In this way, very thin sections (2 μm thick) could be cut through the cubes to allow floc internal structure to be viewed under a microscope. However, when compared to the

agar technique smaller flocs were seen suggesting the hardening resin has a considerable affect on floc structure. Indeed, for both techniques there must be some question as to whether you get random orientation of flocs in the thin agar suspension of the Petri dish.

The use of wide-mouthed pipettes is a common method for floc extraction (Li and Ganczarczyk, 1986 and Spicer and Pratsinis, 1996). Individual flocs may be selected in this way or a larger sub-sample of the suspension may be removed. The latter option is preferred and is more widespread because it requires no bias on behalf of the worker for a selection of a whole range of floc sizes. The use of pipettes is a widely used technique, however Manning and Dyer (1999) suggest that they are a destructive method. They explain that their use may account for differences between floc sizes generated from *in situ* and *ex situ* methods. It is therefore useful to compare *in situ* and *ex situ* techniques. Spicer *et al.* (1998) have compared the size of polystyrene-alum flocs generated from a Malvern Mastersizer (a light scattering method discussed later in the review) using three types of sample delivery mechanisms: a) a 5 mL hand pipette, b) a syringe pump and c) a peristaltic pump. The mass-mean floc diameters after 15-20 minutes of flocculation was ~150 μm using the pipette and ~250 μm using the syringe and peristaltic pumps. This would seem to confirm that hand pipettes adversely disrupt flocs. However, the authors discuss that it was actually the result of flocs settling between sampling and measurement by the Mastersizer because the rate of delivery using to the measuring cell is very slow using the hand pipettes and that the pipettes did not adversely affect the floc size.

Microscopy has the advantage of allowing individual particles to be viewed, scrutinised and analysed at high magnification. This allows the researcher to get a feel for the structure of each aggregate under investigation and give a better indication of floc shape and irregularity. In many applications, microscopy is the only method available to find floc porosity and other shape factors. In addition, microscopy is a relatively inexpensive method. Limitations of microscopy are the small depths of field possible with light microscopes such that a particle may be entirely focused due to its 3D structure projecting into the plane of view (Allen, 1997).

A can be seen from Table 3, the recommended 625 individual particle counts per sample is generally not seen for most studies using microscopy to investigate floc size distributions. This suggests that the microscopy work carried out to date should only be used as an indicator of the actual floc size or as a compliment to other sizing techniques because it does not meet the rigorous statistical criteria of the British standard.

Microscopy has been and still is a widely used technique for floc sizing. Sample extraction and preparation is key to gaining accurate knowledge of floc size distributions. Simply transferring a diluted floc sample into a shallow microscope well is the quickest and easiest and most widely used method. Considerable time and effort is required needs to be invested in order to achieve the necessary accuracy and a statistically significant distribution.

4.2 Photography and image analysis

Image analysis is the manipulation of information within an image to turn it into a more useful form whilst digital image analysis is the manipulation of digital images using a computer (Image Pro guide, 2001). For the purposes of this review, where the term image analysis is used this refers to digital image analysis. The basic stages and requirements of performing image processing and analysis are shown in Figure 3. Image analysis usually requires image processing which is the conversion of one image into another. Usually this is done to improve the quality of the image for analysis. For example, if an image does not have a well defined contrast between the object and background a particle may be incorrectly sized due to a blurred boundary between the two (Chakraborti *et al*., 2000).

The main components of a modern image analysis system are an image capture device (usually a close-coupled device (CCD) camera or digital camera) connected to a computer with an image grabber. Computer software is required for the image processing and analysis and a variety of commercial products are available. Image analysis is often combined with microscopy by mounting a CCD camera onto the microscope (Li and Ganczarczyk, 1986; Cousin and Ganczarczyk, 1998; da Motta *et al*., 2001; Kobayashi, 2004). The advent of photography and image analysis has allowed much quicker measurements of an almost inexhaustible number of different floc size measurements to be made from floc samples when compared to traditional microscope methods (Wang and Gregory, 2002). However, it still relies upon an unbiased approach from the microscopist. Image analysis has also been used to monitor floc suspensions *in situ* (Ducoste and Clark, 1998; Chakraborti *et al*., 2000; Bache and Papavasilopoulos, 2004)). Flocs are monitored by capturing images of a stirred suspension by focusing on a plane a short distance (0.3 – 1 cm) behind the wall

of tank containing the suspension. Calibration is achieved by focusing on a graticule suspended into the tank prior to flocculation experiments.

4.3 Light Scattering

As light is passed through a suspension of particles some part of the light is absorbed by the particles whilst some light is scattered. The remainder of the light passes straight through the suspension. The way in which the suspension does this is dependent upon particle size, the nature of the particles and the suspending medium (Farrow and Warren, 1993). In light scattering particle sizing techniques, the measured scattering pattern of an applied laser is compared to the predicted scattering pattern based upon an optical model in order to generate a particle size (Selomulya *et al*., 2001). Lorenz-Mie theory is the classical model for determining particle diameter from light scattering and is the basis for all particle sizing instruments that measure particle size in this way (Black *et al*., 1996). The principle equation for Lorenz-Mie theory is shown in Equation 3.

$$\chi = \frac{\pi m d}{\lambda} \qquad \text{Equation 3}$$

χ is the fundamental parameter for light scattering, d is the particle diameter, m is the refractive index of the particles and the λ is the wavelength of the incoming laser.

The model assumes particles are (1) spherical, (2) the laser illuminates particles uniformly and (3) the laser beams are plane light waves. This theory works well for particles that are smaller than the cross section of the laser beam. However, as particles get larger assumptions 2 and 3 become unreliable. For example, at a typical beam size of 100 μm, the assumptions of plane light and uniform illumination do not

hold at particle sizes above 10-20 μm (Black *et al*., 1996). Fraunhofer diffraction theory is a modified version of Lorenz-Mie theory that takes this into account (Equation 4).

$$X = \frac{\pi dR}{\lambda f} \qquad \text{Equation 4}$$

X is the fundamental parameter for light scattering, d is the particle diameter, R is the radial distance in the focal plane as measured from the optical axis, λ is the wavelength of the incoming laser and f is the focal length of the receiving lens.

It is important to note the Fraunhofer theory does not depend upon the optical properties of the particles in suspension. The theory holds true for all particles except particles with a refractive index approaching 0 or for very small particles (less than 10 μm). Fraunhofer theory considers only the light diffracted by the particles in suspension, however where a significant amount of light is transmitted through the particles or past the particles the transmitted light (also known as anomalous scattering) impacts on the results.

The most common commercial particle size instruments use light scattering to determine particle size. These instruments (such as the Malvern instruments) measure particle size by passing a laser beam through a suspension of particles. Small particles scatter light at high angles whilst large particles scatter at low angles. An array of ring detectors records the intensity of the scattered light at a range of different angles. From these responses, proprietary computer programmes iterate particle size distributions from Lorenz-Mie and Fraunhofer theory.

A certain amount of light passes through the suspension and out of the optical system. The amount of light lost is dependent upon the size and concentration of the particles in suspension. The instrument measures this in terms of the laser obscuration, in other words the amount of the laser that is scattered or absorbed by the particles. The manufacturers recommend that between 10-30 % obscuration is achieved for reliable measurements (Guan *et al*., 1998). The concentration required to reach this range will vary for different suspensions, however Farrow and Warren (1993) suggest that solids should be < 0.03 % solids by weight. Excessive obscuration leads to significant underestimation of the laser scattering.

These techniques rely upon a constant flow of the suspension through the instrument during the measurement cycle. This feature has been harnessed to allow the development of a non-intrusive methodology for measuring dynamic floc size (Spicer *et al*., 1998; Biggs and Lant, 2000; Chaignon *et al*., 2002). These methods have a stirred vessel containing the aggregate suspension and are connected to the particle sizing device by plastic tubing. Intrinsic to this type of system is a requirement to pump the suspension through the optical unit of the size analyser. As has been previously been discussed, Spicer *et al.* (1998) compared 3 types of pumping techniques for delivery to the optical cell. They concluded that a continuous recycle using a peristaltic pump on the return side of the particle was the least severe technique on the flocs and allowed easy continuous monitoring of the suspension. Flocs were extracted from the recirculation zone in the flocculation vessel (a distance midway between the top of the tank and the stirrer) to ensure an accurate representation of the bulk suspension was sampled. Rattanakawin and Hogg (2001)

used a similar set-up without the recirculation system. The floc samples were discarded once they had passed through the size analyser. This was done to ensure that any affect of the pump on floc size was ignored. However such a method could not be used for continuous monitoring of the flocculation process as the suspending fluid would run dry.

Biggs and Lant (2000) compared the effect of pump speed on the size distribution of activated sludge flocs for a continuous system using a Malvern mastersizer as the size analyser. They compared four different flow rates ranging from 1.7-5.5 ml s^{-1} using a peristaltic pump. They found that the optimum pump speed was at a flow of 3 ml s^{-1}, above this rate shear within the pump and tubing significantly reduced the floc size. Whilst below this flow there was a significant time lag before a representative sample was measured in the particle size analyser. Spicer *et al.* (1998) also used a flow of 3 ml s^{-1} in 6 mm internal diameter tubing (corresponding to a Reynolds number of 618) for research into flocculation of polystyrene beads. Lartiges *et al*. (1994) used a pump speed corresponding to 1.75 ml s^{-1} as this did not encourage aggregation or break-up in the pump system for research on optimising coagulation of raw river water.

4.4 Transmitted light

Another technique that has been used extensively to monitor the size and growth of floc suspensions is the photometric dispersion analyser (PDA). First described by Gregory (1985), the PDA gives a combined measurement of the particle size and frequency for a flocculating suspension. The device consists of a light source, detector

and processing equipment that monitors turbidity fluctuations in the sample. Specifically the PDA measures the average light transmitted through a suspension and the rms value of the fluctuating component (Gregory and Dupont, 1997). The ratio of the two gives the flocculation index (FI), which gives a good measurement of aggregation. The PDA has been widely used in closed loop systems to measure dynamic floc size, similar to those mentioned for the light scattering techniques (Burgess and Phipps, 2000; Fitzpatrick *et al.*, 2003; Yukselen and Gregory, 2004; McCurdy *et al*., 2004). Whilst the other sizing methods give an absolute value for floc size, the PDA gives a combined value that increases with increasing particle size and number. However, for a system containing a constant solids fraction an increase in the FI can be attributed to an increase in floc size as the larger particles have a greater signal fluctuation than smaller ones (McCurdy *et al*., 2004). The PDA is therefore good at showing relative changes in floc size, such as during floc growth and breakage phases and giving qualitative comparisons between different treatment variables. Unlike, some of the other sizing techniques, the PDA is unable to give information on floc size distributions and the solids passing through the measuring cell must be at a high enough concentration to provide a reliable signal.

4.5 Individual Particle Sensors

Individual particle sensors measure single particles as they pass through an aperture onto an electric field (**electrical sensing**) or through a light beam (**optical sensing**). In both of these techniques, the major source of problems for particle sizing comes from

the break-up of floc aggregates as they pass through the aperture of the measuring cell.

In the electric field method, particles are suspended in an electrolyte solution and then passed through an electric field. The change in resistance caused by the particle is proportional to the particle size with a small correction factor (Farrow and Warren, 1993). The Coulter counter is the most common electrical sensing technique. Leentvaar and Rebhun (1983) summarise that the Coulter counter significantly underestimates floc size when compared to optical analysis as it only measures the volume of the solid in the floc and not the effective volume of the floc including pores and water. The effect of the electrolyte solution on floc macrostructure has yet to be fully investigated at the ionic concentrations involved using electric field methods, but Cousin and Ganczarczyk (1998) have shown an increase in activated sludge floc porosity, diameter and elongation at very high salt concentration.

The optical sensing methods measure particles of a size >10 μm. The amount of light attenuated by particles as they cross a light beam is proportional to particle size. However this method is limited by a need for a low particle concentration for accurate measurement and a narrow size distribution band due to the narrow size of each aperture in these instruments and there are few examples of these instruments being used for floc sizing.

4.6 Summary

No particle sizing method is perfect for measuring floc size and each of the techniques have their own advantages and disadvantages. The tedious requirements for sample preparation and floc transfer indicate that microscopy is not the most suitable

technique for finding floc size. Photography *ex-situ* gives a considerable improvement in that flocs do not need removing from the flocculating vessel but high quality images are required for accurate image analysis. Nevertheless, both photography and microscopy can give a crucial feel to the researcher of the type of particle they are dealing with and are often the only reliable method for getting floc shape factors and porosity measurements. The particle sensing instruments are not ideal due to the problems associated with only being able to measure narrow size bands at a time using electrolyte solutions in the measuring cell. On-line techniques such as those involving light scattering and the PDA allow quick measurements to made and their non-intrusive nature and ability to monitor a wide range of particle size distributions make them ideal for showing quantitative size distributions (light scattering) and qualitative changes in floc size (PDA).

5 Fractal dimension

Since Mandlebrot introduced the concept of fractal theory in the 1970's, the application of fractal geometry is now a well established means of describing the complicated structure of particle aggregates (Gorczyca and Ganczarczyk, 1999, Thomas *et al*., 1999; Selomulya *et al.*, 2003; Chakraborti *et al.*, 2003). Fractal objects may be defined as those objects that:

(1) show self similarity

(2) express a power-law relationship between two variables

(3) can be characterised by a non-integer fractal dimension

Self similarity is the existence of the same pattern regardless of the magnification from the which the fractal object is viewed from. In many systems exact self-

similarity is not observed, but a less restrictive definition of a fractal object is that it shows statistical self-similarity. This means that on average different sections of the object look similar to one another (Kaye, 1989). The second characteristic of fractal objects is a power relationship between two variables of the object. This may be a link between area (A) and length (L) as in Equation 5, or the relationship between volume (V) and area as in Equation 6.

$$A \propto L^{D_f} \qquad \text{Equation 5}$$

$$V \propto A^{D_f} \qquad \text{Equation 6}$$

Flocculated aggregates are examples of mass fractal objects. This means that both the internal structure and the surface of the aggregate exhibit fractal properties. Mass fractals are summarised by Equation 7.

$$M \propto L^{D_f} \qquad \text{Equation 7}$$

M is the mass of particles, L is a characteristic measure of size and D_f is the mass fractal dimension. Gregory (1998) summarises that the choice of measurement for the size L does not matter as the same trends are seen so long as the choice is constant. For Euclidean objects, the one dimensional value of D_f will be 1 for a linear line, 2 for a two dimensional planar shape and 3 for a compact three dimensional shape. Fractal objects take non-integer values of D_f and are therefore said to show non-Euclidean dimensionality. Values approaching 3 for a three dimensional floc therefore indicates a high degree of compaction whilst values approaching 1 indicates a very loose and open structure. The fractal dimension can therefore give important structural information of floc compaction and the space filling nature of the aggregate. The fractal dimension of a floc may be found in a number of ways. These may be broadly

categorised into techniques that use scattering (of light, neutrons or x-rays), settling and two dimensional fractal analysis using image analysis (Waite, 1999).

5.1 Scattering

The pattern in which an aggregate scatters incoming radiation gives information on the aggregate structure as a function of a length scale (Bushell *et al*., 2002). The way in which an object scatters light can give fractal values if enough is known about the scattering properties of the material contained within the aggregate. This technique assumes:

(1) The primary particles that make up the aggregate are uniform in shape and size.

(2) The refractive index of the aggregate material is low so that the wavelength of the incident light does not become shortened.

(3) Light is only scattered once as it passes through the suspension of aggregates before hitting the detector. Multiple scattering should be minimised by ensuring the concentration of particles is low (Tang *et al*., 2002).

Generally, finding the fractal value from scattering theories rely upon the power law based upon Rayleigh-Gans-Debye (RGD) scattering theory shown in Equation 8.

$$I(Q) \propto Q^{-D_f} \qquad \text{Equation 8}$$

I(Q) is the intensity of the scattered radiation and *Q* is the wave number estimated from Equation 9.

$$Q = \frac{4\pi n \sin(\theta/2)}{\lambda} \qquad \text{Equation 9}$$

n is the refractive index of the suspending medium, θ is the scattered angle, λ is the wavelength of the radiation in a vacuum.

The fractal dimension D_f is found from the slope of the line of a log-log plot of $Q(I)$ against Q (Wu *et al*., 2002). For fractal objects a power law relationship exists between $Q(I)$ and Q. This dependency is only valid when:

$$\frac{1}{R_{agg}} \rangle\rangle\ Q\ \rangle\rangle\ \frac{1}{R_{part}} \qquad \text{Equation 10}$$

R_{agg} is the radius of the aggregate and R_{part} is the radius of the primary particle.

This is because when Q approaches the size of R_{agg} the relationship is affected by the edges of the aggregate whilst when Q approaches the size of R_{part}, light is mainly scattered by the primary particles and not the aggregate (Guan *et al*., 1998; Waite *et al*., 2001). The primary particles of the flocs must also satisfy independent scattering for RGD scattering theory approximations to be obeyed. The RGD approximation is deemed applicable when:

$$|m-1| << 1 \qquad \text{Equation 11}$$

$$(2\pi m/\lambda)L|m-1| << 1 \qquad \text{Equation 12}$$

where m is the material refractive index and L is the length of the scattering body.

Most application of scattering has been to mono-disperse systems where information is known about primary particle size and the scattering behaviour of the particles

under investigation. Examples include flocs formed from particles of latex (Tang, 1999; Selomulya *et al.*, 2001), aluminium oxide (Waite *et al*., 2001), and iron oxyhydroxide (Waite, 1999). In these cases the assumptions mentioned above are generally valid for application to floc aggregates. Application to more complex flocs typically found during water and wastewater treatment processes has been more difficult. In these instances, information on primary particle composition can be limited with little to no knowledge of particle refractive index. Furthermore the primary particles may be non-uniform and consist of particles with different refractive indices. Therefore a number of the assumptions mentioned above may not be met when analysing complex floc structures. As a practical example of this, a mixture of iron oxyhydroxide and kaolin has a considerably distinct shaped scattering curve when compared to a pure iron oxyhydroxide system (Waite, 1999). In the case of activated sludge good scattering law relationships have been seen (Guan *et al*., 1998; Waite, 1999). This is because the very low refractive index of the bacteria in the floc allows RGD theory to be met.

A common limitation to this technique is the limited scale of investigation of the technique. Commonly, the scattering power law relationship breaks down at small floc sizes compared to the average floc size. This was seen for kaolin suspensions where the average floc sizes ranged between 200-350 μm in diameter whilst the linear portion of the linear scattering relationship applied to flocs less than 50-100 μm (Wu *et al*., 2002). For activated sludge flocs ranging up to 400 μm in diameter, the power law cut-off was below 70 μm (Waite, 1999). The application of light scattering to larger floc systems is an area that needs further investigation to find whether the larger flocs have variable fractal dimension or interfere with the scattering.

5.2 Settlement

Using settlement as a means of determining the fractal value of aggregates is a well established technique to the more widely used small angle light scattering method. The use of sedimentation to determine floc structural characteristics takes on an extra relevance because the settling behaviour of aggregates is an important parameter for optimising the sedimentation procedure. Floc settling behaviour is dependent upon size, effective density and porosity (Tang *et al*., 2002). The fractal structure of flocs structure can have two possible consequences on its settlement behaviour because flocs take on increasingly non-spherical forms as they grow. This may act to increase the drag on the particle when compared to a solid sphere of the same size. Conversely, the porosity of flocs can act to reduce drag by allowing advection of the suspending medium through the floc structure (Bushell *et al*., 2002).

The following determination of floc fractal dimension from settling velocity has been taken from Miyahara *et al*. (2002). A spherical particle at its terminal settling velocity may be summarised by Stoke's law as shown in Equation 13.

$$v = \frac{(\rho_s - \rho_l) g d}{18 \mu} \qquad \text{Equation 13}$$

v is the terminal settling velocity, ρ_s is the density of the particle, ρ_l is the density of the liquid, d is the floc diamater, μ is the viscosity of the suspending medium and g is acceleration due to gravity. Whilst Stokes law may be an over-simplification to completely describe floc setting, it is generally believed that flocs settle slow enough in order for Stokes derived equations to apply (Gregory, 1998). Shape factor and drag

coefficient corrections are usually added to account for the irregular shape of flocs. A fractal floc consisting of similar primary particles may be summarised by:

$$i = \left(\frac{d}{d_p} \right)^{Df} \qquad \text{Equation 14}$$

where i is the number of primary particles, d_p is the primary particle diameter and D_f is the fractal dimension. The mass and volume balances of the floc are given by:

$$V_f = V_s + V_l \qquad \text{Equation 15}$$

$$\rho_f V_f = \rho_s V_s + \rho_l V_l \qquad \text{Equation 16}$$

Where V_f is the floc volume, V_s is the volume of solids in the floc and V_l is the volume of liquid in the floc. Combining equations 14-16 into 13 gives:

$$v = \frac{d_p^{3-D_f} d^{D_f - 1} (\rho_s - \rho_l) g}{18 \mu} \qquad \text{Equation 17}$$

The slope from a log-log plot of floc settling velocity against size will therefore yield the fractal dimension (Johnson *et al.*, 1996). The fractal dimension is found from the slope of the plot with D_f being equal to the value of the slope + 1. The equation only applies when the floc Reynolds number is less than one and the flocs are fall isolated at their terminal settling velocity in laminar floc. Wu *et al.* (2002) summarise that most experimental systems measuring settling rate meet the criteria for finding the floc fractal dimension. Problems may be accounted when the floc porosity is high, in these instances advection floc through the floc significantly increases the settling rate

over that predicted by Stokes equations. In most practical instances, porosity effects on settling are neglected and currently far from understood. Therefore, care must be made of interpreting data when the floc fractal dimension is significantly less than 2 (Gregory, 1998). In addition, measuring floc settling requires meticulous preparation and a large sample number in order to get accurate results (Bushell *et al*., 2002).

5.3 Image analysis

The combination of microscopy and image analysis software have has been widely used to floc fractal dimension (Bellouti *et al*., 1997; Cousin and Canczarczyk, 1998; Chakraborti *et al*., 2003). Generally, high quality images of flocs are taken and the two dimensional (2D) fractal dimension found. This may be achieved in two common ways. The first is from the relationship between floc area and length (Equation 5). A log-log plot of floc area against size as found from image analysis yields a line with a slope giving the fractal dimension (Chakraborti *et al*., 2000). A second way of determining floc fractal dimension is the box counting method. The process begins by covering the floc image with boxes of a minimum size to just cover the floc, this is then repeated with smaller box sizes (Bushell *et al*., 2002). Plotting the number of boxes needed to cover the object against the size of the box on a log-log scale gives a line with a slope equivalent to the fractal dimension (Bellouti *et al*., 1997). Commercial software packages are able to do this analysis very quickly and easily.

The main requirement for 2D fractal dimension analysis using image analysis is for the image to be of suitable quality for commercial software packages to be able to distinguish the floc from the background (Chakraborti *et al*., 2000). In practice this often requires considerable image correction prior to fractal analysis and works best with flocs that show good contrast with their background and are not transluscent.

5.4 Summary

To summarise, Table 4 highlights the major advantages and disadvantages of each of the techniques mentioned. Light scattering methods work well with small, open flocs that have low refractive indices. Bushell *et al.* (2002) state that these are precisely the type of aggregates that settlement and image analysis techniques do not apply well to because they do not settle well, the modelling of the settling of open flocs is very difficult due to permeability effects and these particles are difficult to see because of their low refractive index. However, when analysing systems of high particle concentrations, shadowing effects and multiple light scattering invalidates the scattering models. Therefore, the calculation of fractal dimension and particle size only holds when within the obscuration threshold of the scattering instrument being used (Guan *et al.*, 1998). Similarly, flocs composed of a number of different primary particles have scattering behaviour that is difficult to predict and should not be used in these instances. The technique is also limited in that fractal values are generally only found for the small flocs in the system. However, when using the settling technique the fractal relationship is always seen across the whole range of flocs under investigation. Settling is a reliable technique provided a large enough sample is measured which can make this technique very time consuming. Settling can be widely applied to most floc systems provided that flocs are relatively compact thus avoiding interference in settling from porosity and advection though the floc. Careful temperature control and quiescent conditions in the settling column must be provided in order to prevent disruption to the floc.

2D image analysis relies upon flocs that have a high degree of definition between the solid of the floc and the background. Therefore pale translucent activated sludge flocs

are often not ideal for this technique. In addition sample preparation prior to image capture is an important consideration and as has been mentioned in previous sections, the preparation stage must not act to damage or interfere with the floc structure. The main advantage of the box counting method is that the fractal dimension of individual flocs are measured. This can highlight differences in floc fractal dimension within a system whilst all the other techniques report average fractal values for the whole system.

Finally, it is important to report which technique has been used to measure floc fractal dimension. This is because each technique gives a different answer and may in fact be measuring different structural properties of the floc. For example, in a comparative study by Wu *et al*. (2002) the fractal dimension of activated sludge using settling was 1.31 whilst it was 2.06 using light scattering. Furthermore, image analysis of 2D images can only give a maximum fractal value of 2 whilst the maximum is 3 for the analysis of 3D flocs in settling and scattering.

6. Overall summary

A range of techniques for measuring floc structural characteristics of size, shape and fractal dimension have been presented. Of the sizing techniques, microscopy is the most time consuming requiring considerable sample preparation and analysis time in order to achieve satisfactory results. However, microscopy and photography can give an important feel for the type of floc under investigation and is also the only reliable method to determine floc shape characteristics. On-line light scattering and PDA devices show good capability for measuring a whole range of different floc types and size in a non-intrusive way. Fractal dimension analysis can be measured using three

main techniques. Light scattering works best for small, open flocs of low refractive index whilst larger, flocs of low porosity and of high colour contrast are more suited to settling and 2D image analysis under a microscope.

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Figures

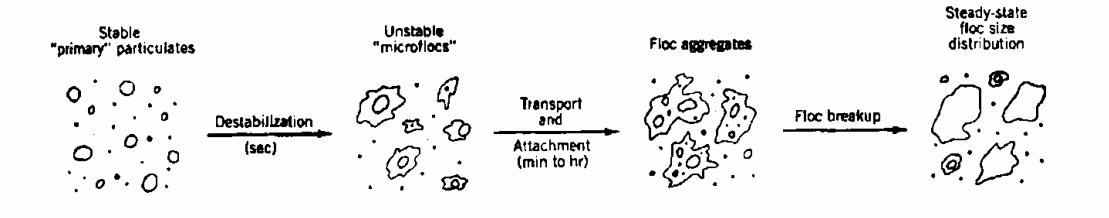


Figure 1.

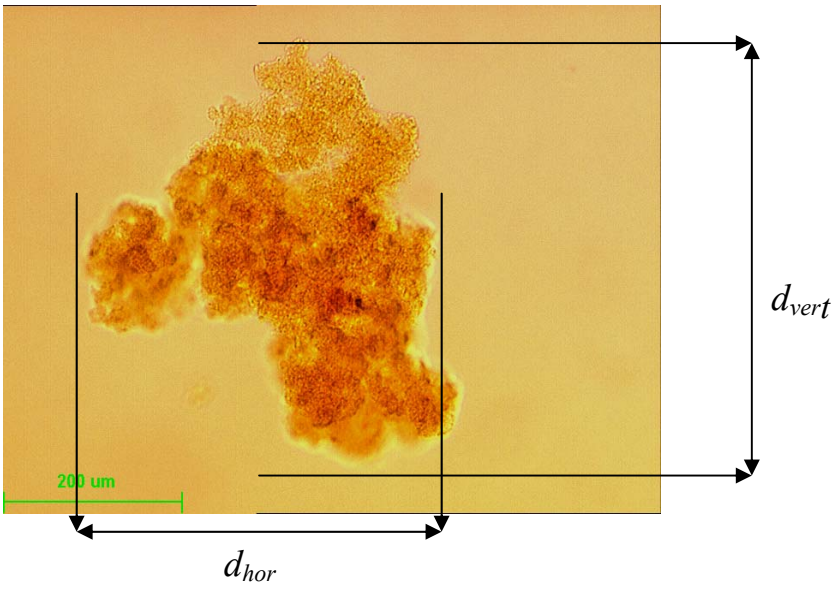


Figure 2.

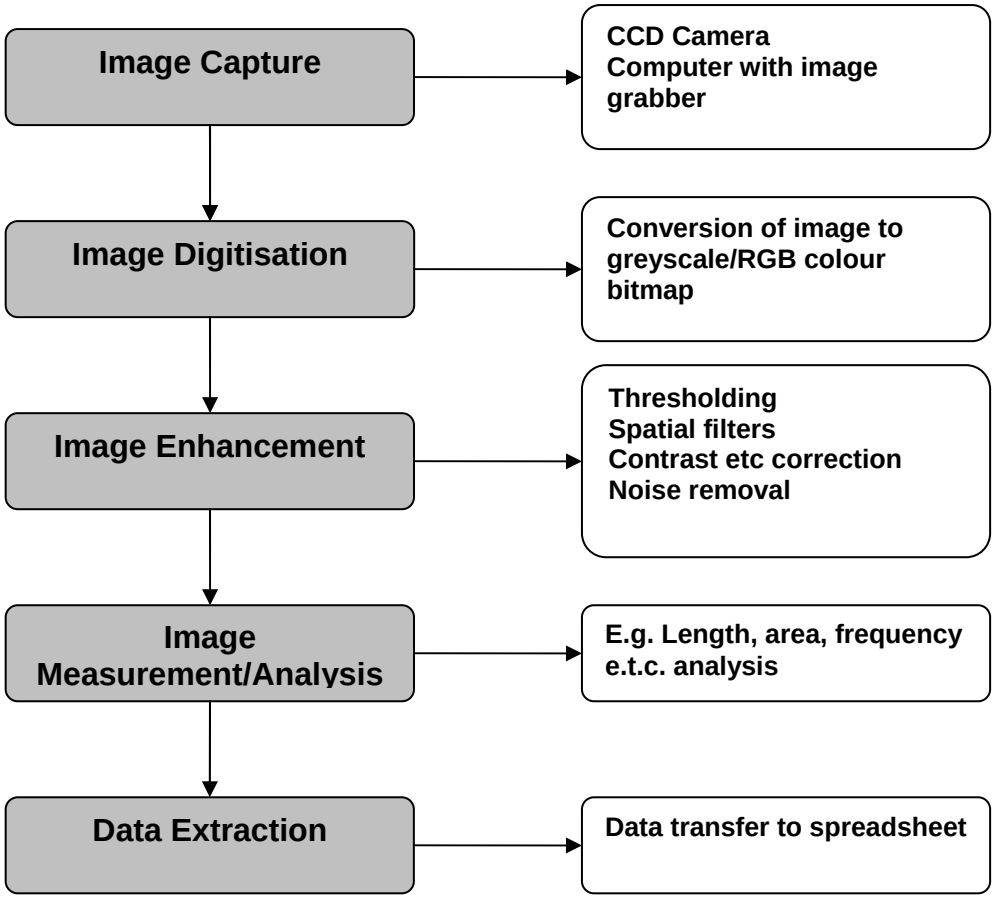


Image Capture
CCD Camera
Computer with image grabber
Image Digitisation
Conversion of image to greyscale/RGB colour bitmap
Image Enhancement
Thresholding
Spatial filters
Contrast etc correction
Noise removal
Image Measurement/Analysis
E.g. Length, area, frequency e.t.c. analysis
Data Extraction
Data transfer to spreadsheet

Figure 3.

Figure legends

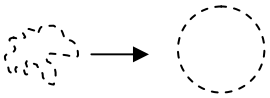
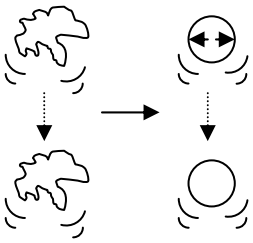
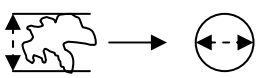
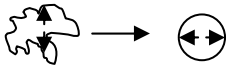
Figure 1. The steps of particle transport and attachment for aggregating particles (from Montgomery, 1985).

Figure 2. The maximum dimensions in the horizontal and vertical planes for a typical floc.

Figure 3. The steps involved in digital image analysis

Tables

Table 1. The most common equivalent diameters used for characterising floc aggregates (taken from Dharmarajah and Cleasby, 1986 and Allen, 1997).

Floc Diameter	Description	Diagram	Equation for Calculation
Perimeter diameter, d_c	The diameter of a circle with the same perimeter (*P*) as the measured particle.		$d_c = \frac{P}{\pi}$
Projected area diameter1, d_a	The diameter of a circle with the same projected cross-sectional area (*A*) as the floc measured in a stable orientation.		$d = 2\sqrt{\frac{A}{\pi}}$
Projected area diameter2, d_p	The diameter of a circle with the same projected area as the floc measured in a random orientation.		
Surface diameter, d_s	The diameter of a sphere having the same surface area (S) as the floc.		$d_s = \sqrt{\frac{S}{\pi}}$
Volumetric diameter, d_v *(OR equivalent spherical diameter)*	The diameter of a circle with the same volume (*V*) as the floc measured.		$d_v = \sqrt[3]{\frac{6V}{\pi}}$
Surface-volume diameter, d_{sv}	The diameter of a sphere with the same surface area to volume ratio as the floc.		$d_{sv} = \frac{d_v^3}{d_s^2}$
Free-falling diameter, d_f	The diameter of a sphere having the same density and free-falling speed as the floc in the same fluid at the same density and viscosity.		
Stoke's diameter, d_{st}	The diameter of a free falling particle in the laminar flow range (where Re < 0.2).		$d_{st} = \frac{18\mu v}{\rho_f - \rho}$
Feret's diameter, d_F	The (mean) value between pairs of parallel tangents to the projected outline of the particle.		-
Martin's diameter, d_M	The length of the chord parallel to a fixed direction which splits the floc projected area into two equal parts.		-

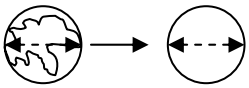
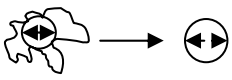
Circumscribing diameter, d_{sc}	The diameter of the smallest circle that circumscribes the outline of the projected floc.		-
Inscribing diameter, d_i	The diameter of the biggest circle that fits inside the outline of the projected floc.		-

Table 2. Some of the methods used for obtaining aggregate size.

General Floc Sizing method	Floc Size found from:
Microscopy	(1) observation of static floc size (2) observation of dynamic floc size
Photography and image analysis	(1) observation of floc static size taken from suspension (2) observation of floc dynamic size under turbulent conditions (3) observation of floc dynamic size under laminar flow
Light scattering	(1) back/front scattering of light by floc particles
Transmitted light	(1) light transmitted through floc suspension
Individual particle sensors	(1) optical sensing of flocs (2) electrical sensing of flocs

Table 3. The number of flocs sized per sample for a number of different studies.

Type of floc under investigation	Number of flocs measured	Authors
Activated sludge	245-377	Li and Ganczarczyk (1986)
Polystyrene beads	500+	Spicer and Pratsinis (1996)
Activated sludge	70+	da Motta *et al*. (2001)
Alum flocs	~ 100	Gorczyca and Ganczarczyk (1999)

Table 4. The advantages and disadvantages of the techniques used for determining the fractal dimension of floc aggregates.

Technique	Advantages	Disadvantages
Light scattering	- Rapid, non-intrusive method - Lends itself well to dynamic, online analysis - Very good for analysis of small aggregates with an open structure and low refractive index - Takes a large number of readings from many aggregates in a few seconds	- Not good for polydisperse aggregates made from many primary particles - Choosing an appropriate model for scattering behaviour can be difficult - Results affected by contamination from dust etc. - Power-law relationship breaks down at large floc size
Settling	- Best for measuring fractals of compact flocs - Cheap and simple - Not prone to contamination issues - Good for aggregates of made from a number of different primary particles	- Time consuming - Finding an appropriate drag coefficient is difficult - Can get non-random orientation of falling aggregates - Careful regulation of settling column required
Image analysis	- Best for large, open aggregates - Not prone to contamination issues - Examination of single flocs allows detailed information on variation in floc structure within a sample	- Time consuming - Requires well defined, high contrast images for accurate analysis – which flocs generally aren't

https://dspace.org/

Measuring Floc Structural Characteristics

Jarvis, Peter; Jefferson, Bruce; Parsons, Simon A.

2005-05
Springer
http://hdl.handle.net/1826/985
Jarvis, Peter; Jefferson, Bruce; Parsons, Simon; Measuring Floc Structural Characteristics. Reviews in Environmental Science and Biotechnology. Vol 4 (1-2), May 2005, pp. 1-18

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