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Loading of Solids in a Liquid Medium: Determination of CBVC by Torque Rheometry

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Abstract

In many applications involving solid–liquid mixtures such as powder injection moulding, it is necessary to know the minimum amount of liquid necessary to provide fluidity to the powder–liquid mix. This quantity, referred to as critical binder volume concentration (CBVC), can be determined for a given powder by torque rheometry. This paper discusses the optimization of various parameters involved in the determination using torque rheometry, along with the application of the method to a variety of powders of different sizes and shapes. The CBVC value thus determined represents the minimum void volume achievable for a given powder, and thus can form the basis for deciding the amount of binder desired for injection moulding.

1 Introduction

Filling of solids in a liquid medium finds a variety of technological and commercial applications, from the formulation of common paints and pigments, to fabrication of high-tech ceramic products of complex shapes by injection moulding. In all these applications, it is useful to know the optimum quantity of the liquid medium required to disperse and mix the solids thoroughly with the liquid. Despite this, not much attention has been paid to the systematic study of this process so far. Literature records only about a dozen papers over past several decades.[1–17]

From the literature, it appears that the first sector to address this issue was the paint industry. In the early 1920s, it was noticed that the properties of a pigment–thinner mixture of a paint formulation changes drastically beyond a critical amount of solid in the liquid. Gardner and Coleman[1,2] proposed the concept of oil absorption value as an estimate of this quantity, and described a procedure for determining the same experimentally (ASTM-D1483). A slightly modified procedure, widely known as Spatula Rub-Out test,[3] was tentatively adopted in 1927 as the standard, ASTM D-281. This method was considered superior to the former one in view of the additional dispersion achieved during the rubbing operation. Klumpp[5] proposed that the oil absorption value measured is related to the pore or void volume of a closely packed pigment. However, Gardner and Coleman[1] and Baldwin[6] thought it to be simply a function of the specific surface area of the pigment.

These divergent views gave way to a somewhat coherent approach in the late 1940s with the development of the concept of critical pigment volume concentration (CPVC). According to this concept, developed largely by Asbeck and Van Loo,[7] CPVC is the pigment content, expressed as vol% pigment in the pigment–binder mix, at which the binder is just sufficient to form a strong, adsorbed layer on the pigment particles and to completely fill the inter-particulate voids (see Fig. 1). It represents the densest packing of the pigment particles commensurate with the degree of dispersion of the system. Once the pigment volume becomes more than the CPVC, the properties of the powder dominate that of the mix. Similarly, if the pigment volume is less than the CPVC, the properties of the mix are determined largely by those of the liquid.

Thus, the above definition provided a conclusive physical interpretation to the set of experimental observations of paint film properties, such as gloss, corrosion resistance, permeability, hiding power, washability, etc.[8] And, PVC/CPVC ratio became an important factor in formulating paints with desired properties. This concept can be easily extended to all situations where a powder—be it an organic or inorganic pigment, or a ceramic or a metal powder—is required to be intimately mixed with a liquid in any particular usage. The acronym CPVC can be then be suitably altered to mean critical particle volume concentration.

In many of the practical applications, the liquid

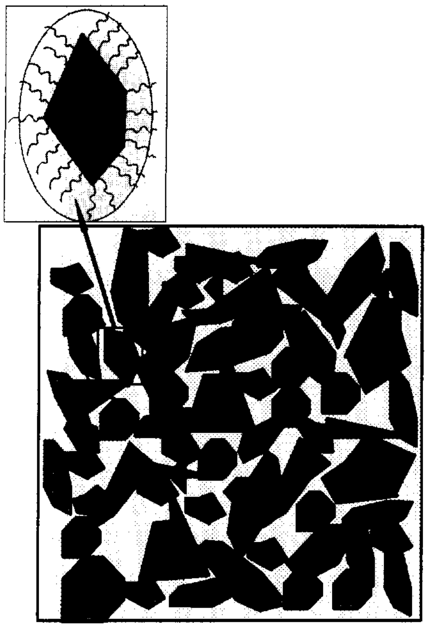


Fig. 1. Schematic particle structure at the point of CBVC showing the interparticulate voids completely filled by the binder liquid; the inset shows the adsorbed liquid forming a boundary layer around a particle.

used acts essentially as a binder for the particulate system, and the need is to know the volume of this liquid to be added. Keeping this in view, it is preferable to use the above concept in terms of its complement, the critical binder volume concentration (CBVC). In fact, the oil absorption value measured experimentally is the CBVC value itself.

In spite of the practical significance of CBVC, the Spatula Rub-Out test[3] remained the only popular method for its determination until the late 1950s. According to this method, a known weight of the pigment sample is kept on a glass plate or a marble slab. Raw linseed oil is added drop by drop to this pigment, and rubbed with a sharp edged steel spatula so as to incorporate the oil in the powder completely. The end point of the test is when a very stiff, putty-like paste, which does not break or separate on rubbing, is formed. This method is poorly reproducible and highly inaccurate because of the human subjectivity involved in the determination of the end point.

Bessey and Lammiman[9] used a density measuring technique to estimate the CBVC. In this, the powder is mixed with the binder in varying proportions, compacted and the density determined. The mix showing the peak density corresponds to CBVC. Warren and German,[10] Pujari[11] and others[12] have applied this technique to ceramic and metal powders. Hay[13] suggests that this technique is good only if complete removal of air, proper dispersion to eliminate all agglomerates and tightest possible packing of the particles is ensured. While these conditions are met easily for mixes containing binder in excess of CBVC, it is not so for sub-CBVC compositions.[14] The resulting error in densities for these samples makes the determination of the point of peak density highly subjective and inaccurate.

Wirsching and Haug[15] proposed the first instrumental method based on measuring the torque as a function of the binder content using a low shear, sigma type torque measuring mixer. Although this method is far superior to the Spatula Rub-Out test, it remained largely unknown and unexplored except for some specialized purposes.[16,17] Hay[13] re-investigated the method for the determination of oil absorption value of pigments using a Brabender Plastograph. Markhoff *et al.*[18] applied this technique for the estimation of CBVC of ceramic mixes used for fabrication of high-tech components by ceramic injection moulding. Pujari[11] proposed an alternative method based on the mechanical exotherm observed during the mixing of a molten binder with a powder in a Helicone mixer having two intermeshing blades.

Pujari's method involves a series of mixing experiments with different powder-to-binder ratios in order to estimate the CBVC, and hence is laborious and time consuming. In addition, the mechanical exotherm, which is attributed to the change of mixing from distributive to dispersive mode, was not observed in our repeated experiments in the Banbury type mixer of the Brabender Torque Rheometer. In both the work of Hay[13] and Markhoff *et al.*[18] the variation of the torque, as a function of the oil volume added to a known volume of the powder kept in the mixing bowl, is measured. A sharp change in the torque indicates the end point. In the above studies, however, the experiments were confined to only very fine powders, and limited volumes and single mixing speed. The effect of various instrumental parameters, as well as the role of particle size and shape of the powder used were not investigated in detail. This has limited the general applicability of this versatile technique for determining the CBVC of a variety of powders. This paper presents the result of our comprehensive studies on these aspects of the experimental methodology.

2 Experimental

The instrument used is a Brabender PL-2000-3 Torque Rheometer fitted with W 50 E mixing bowl and roller blades. Linseed oil of acid value 2·5 is required for mixing.

The general procedure is as follows. The torque range of the torque rheometer is calibrated for a set rotational speed (between 50 and 100 rev min^{-1}) of the blades. About 50 ml (apparent volume) of the powder is accurately weighed. Half this powder is filled into the mixing bowl and 5 ml of linseed oil is added. After mixing for about 5 min, the balance powder is added and the mix is titrated with linseed oil. During the experiment, the oil is added to the bowl, either continuously or stepwise, using a burette. The torque is recorded continuously as a function of time using the computerized data acquisition system. The experiment is stopped after the torque value has passed through the peak value and dropped substantially. The volume of the binder corresponding to the peak value of the torque is used to estimate the CBVC.

3 Results and Discussion

3.1 Principles

The process of mixing in a Banbury type mixer, along with a typical torque vs. time curve are illustrated in Fig. 2. When an aliquot of a liquid is added to a powder mass, the torque rises sharply initially and then starts falling until it attains some stable, mean value as shown in Fig. 3 (a). The initial sharp rise is due to the immediate immobilization of the liquid by the powder particles to form a few big clusters. The immobilized liquid proceeds either to form an adsorbed liquid layer on the particles, or to bind the particles together by liquid bridging, or to fill the voids within the cluster. Both liquid bridging and void filling lead to the formation of very tight clusters which are usually tough because of the strong surface tension force involved. Considerable stresses are therefore required in order to break these clusters. Thus, during mixing at a constant shear rate, high torques are encountered initially when only a few big clusters are present. As the shearing action is continued, these big clusters break down into a large number of smaller ones, until some equilibrium distribution is reached. Corresponding to this, the torque also shows a downward trend and, ultimately, stabilizes at a certain level depending on the amount of the liquid added. If a small aliquot of the liquid is again added to this solid–liquid mix, a similar but smaller torque rise and stabilization occurs, as shown in Fig. 3(b). The mean equilibrium size of the clusters at the end of such an equilibration cycle will depend inversely on the shearing rate used (i.e. the rev min^{-1} of the mixing blades), and directly on the amount of liquid available for binding the particles.

As we continue adding the liquid, the mean equilibrium size of the clusters becomes bigger and bigger as more and more particles join together, until ultimately the whole mass becomes a coherent paste. Corresponding to this, the torque also continuously increases until it reaches a peak at the point of CBVC. From then on, addition of any further liquid serves only to dilate the solid structure and increase the interparticle distances. Therefore, the mixing torque falls as shown in Fig. 3 (c).

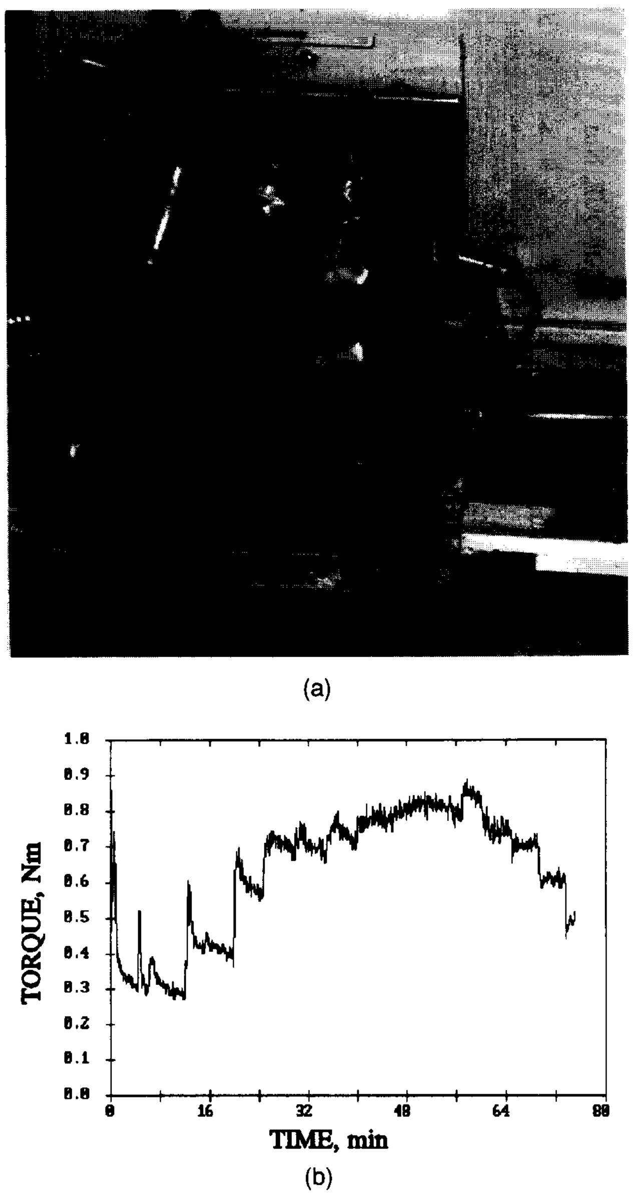
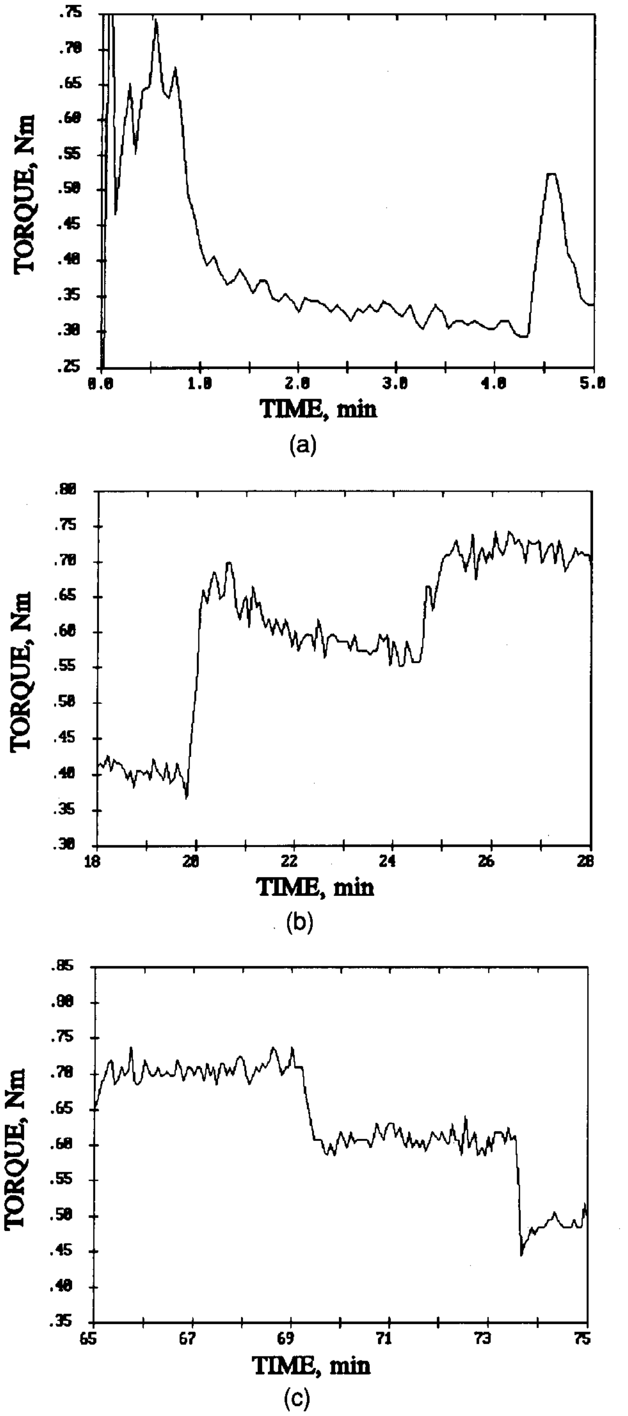


Fig. 2. (a) A Brabender mixer with roller mixing blades having a free bowl volume of 55 ml. (b) Variation of torque during a titration of 65 g of fused silica powder with linseed oil using roller blades rotating at 50 rev min^{-1}.

3.2 Optimization of experimental parameters

In standardizing the method for estimating CBVC using the above principles, various parameters such as the type of the titrant liquid to be used, the amount of powder to be filled into the mixing bowl, the rotational speed of the mixing blades and the manner in which the binder is added to the mixing bowl are to be optimized with respect to the sharpness of the endpoint and a smooth conduct of the titration. For this, two types of powders having different characteristics have been used: fused silica powder has a multimodal



(a)

(b)

(c)

Fig. 3. Variation of torque with time: (a) On first addition of 5 ml of oil; (b) on addition of 0·5 ml of oil at some intermediate stage before the point of CBVC; (c) on addition of 0·5 ml of oil beyond the point of CBVC.

distribution of irregular shaped particles having an average particle size of about 35 μm, while alumina has a narrow monomodal distribution of irregular shaped particles centred around 2 μm.

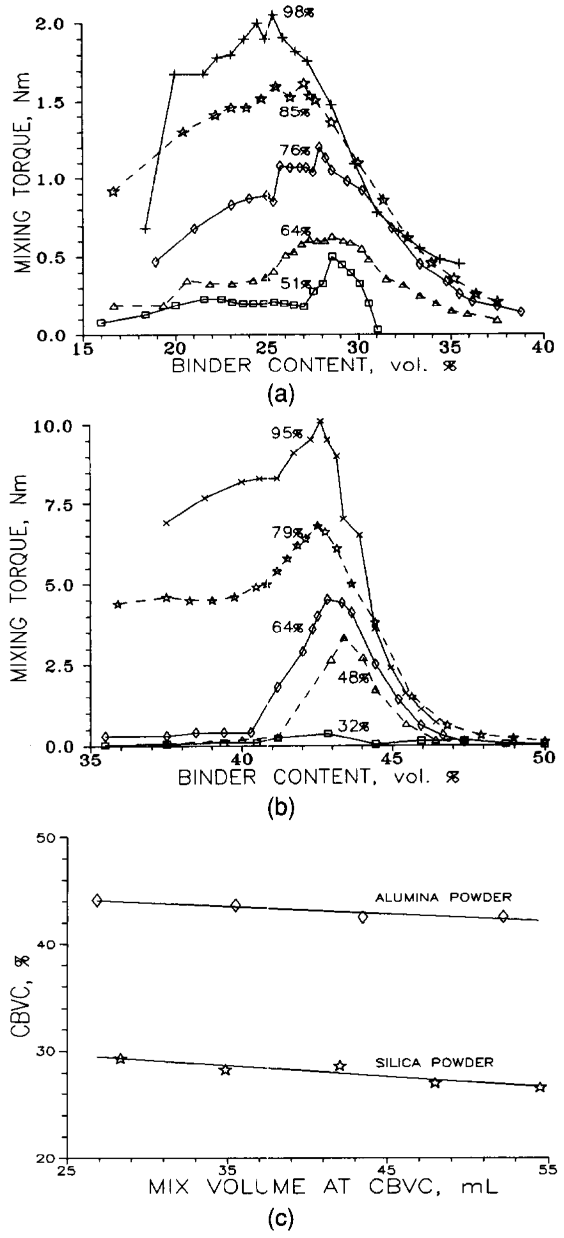
3.2.1 *Choice of the binder liquid*

One of the points implicit in the application of the torque rheometer method is that the binder liquid wets the powder very well, as only a wetting liquid can flow into the interparticulate regions and give rise to interparticle bonding and hence torque. Also, as the liquid is to be added to the powder in the mixing chamber during the course of the experiment either continuously or in small aliquots using a dispenser or a burette, it must have reasonably good fluidity. Higher fluidity also facilitates faster flow into the interparticulate regions, and leads to shorter experimental times. In addition, the wear of the mixing chamber and the mixing blades will be minimized if the liquid is able to lubricate the particles. Thus the choice is essentially limited to low viscosity, wetting type liquids, having some lubricating properties. Many natural oils such as linseed oil, olive oil, sunflower oil, etc., which are multicomponent mixtures, satisfy these requirements. These oils are polar enough to wet most metal and ceramic powder surfaces which are essentially polar in nature, and they are acceptable in terms of their fluidity and lubricating behaviour. It was decided to use linseed oil as it is most commonly used in the paint industry as a vehicle, and hence is inexpensive and easily available.

3.2.2 *Volume of powder*

Hay[13] has suggested the total volume of the pigment and oil to be about 35 to 40 ml in 83·5 ml bowl based on his studies with diatomaceous silica and talc, while Markhoff *et al.*[18] have proposed that a theoretical powder volume of 25 ml for a 55 ml bowl be used. The former does not provide any basis for selecting the initial volume of the powder without trial and error experiments, and the latter is too arbitrary to apply for different powders.

Figures 4 (a) and (b) show the torque vs. binder volume concentration (BVC) curves for different volumes of silica as well as alumina powders. The total bowl volume is 117 ml. Out of this, the roller type mixing blades occupy a volume of 62 ml. The free bowl volume (FBV) available for holding the mix is thus 55 ml. (If cam blades are used for mixing, then the free bowl volume will be 83 ml.) In the graphs, the total volume of the mix is expressed in terms of per cent free bowl volume filled at the point of CBVC (% FBV). The y-axis in the graphs represents the torque value after mixing equilibrium is achieved and the x-axis represents the volume of the liquid added expressed as the vol% oil in the mix. Figure 4 (c) presents the CBVC values for different volumes of the mix at the point of CBVC. It is clear from the figures that, as the amount of the powder used is successively increased, the torque level increases. Except for small volumes, neither the end point definition nor the value of CBVC is significantly affected. But high volume of the powder implies higher mixing times and greater wear of the mixer, and is therefore undesirable. From this, it appears ideal that a volume of the mix sufficient enough to fill at least 50% FBV at the point of CBVC should be used for conducting the mixing experiment.



(a)

(b)

(c)

Fig. 4. Equilibrium mixing torque as a function of binder volume concentration for different volumes of the powder: (a) fused silica; (b) alumina. Roller blades at 50 rev min^{-1} were used. Values near the curves indicate the % free bowl volume filled at CBVC. (c) CBVC data from the graphs (a) and (b) vs. the total mix volume at CBVC.

However, the packing density of free powders imposes a limitation which precludes following this criterion universally. Particles of a powder mass do not flow freely and pack to their optimum level because of interparticle friction. Table 1 presents the volume of a powder pack, having an initial apparent volume of 100 ml on tapping (corresponding to the tap density) and that in the presence of a liquid (corresponding to the point of CBVC). It is clear that packing of powder particles is far more efficient in the presence of a liquid than when just tapping. This is because mixing with a lubricating liquid permits the particles to slide easily past each other and pack. The particle packing is also greatly assisted by the capillary forces due to the presence of the liquid in the interparticle regions.[19] As a consequence, the actual volume occupied by the powder when mixed with a liquid is lower than that in the absence of the liquid, and this difference becomes very pronounced especially for irregular shaped, fine powders.

In view of this, even if the powder is filled in the bowl to the topmost level of the blades in the beginning of the mixing experiment, its volume will decrease once the titration with linseed oil is commenced. For some powders, the volume may not even be sufficient to ensure good mixing by shearing action during the rotation of the blades. In fact, if the powder volume becomes very small, the powder–oil mix simply sticks to the blades and is physically carried along with them, and no mixing takes place. On the other hand, if more powder is forcibly filled with pressure into the bowl to overcome this problem, the oil flowing through the hole in the ram will form an impervious, oil–powder layer at the top of the bowl that does not allow further oil into the bowl. If the mix volume is too high to be accommodated in the bowl, either the shear pins of the blades will break or the mix is simply thrown out of the bowl. It will not be possible to continue the test under either of these situations.

Thus, there is a serious problem in specifying a

Table 1. Packing volumes of different powders

Powder	*Density (g cm^{-3})*	*Weight (g)*	*Apparent volume (cm^3)*	*Volume after tapping (cm^3)*	*Volume at CBVC (cm^3)*
Fused silica	2·18	65·0	100	65	52
Alumina	3·97	99·2	100	83	44
Coarse SiC	3·21	160·0	100	83	75
Fine SiC	3·21	96·0	100	63	54
Zirconia	5·90	112·0	100	65	36
Fine silica	2·18	23·8	100	76	23
Fine zircon	4·70	150·4	100	53	41
Coarse zircon	4·70	126·9	100	56	43
Coarse nickel	8·90	506·4	100	97	91
Fine nickel	8·90	110·3	100	73	53
Coarse alumina	3·97	103·4	100	93	60

priori the amount of the powder that can be filled in the mixing bowl at the beginning of the titration. In order to overcome this, a different approach has been devised, following the data from Table 1 for a variety of powders with different shapes and sizes, and assuming that the maximum decrease in volume in the presence of the oil is about 80%. About 50 ml of the apparent volume of the powder is accurately weighed. Half this volume is added into the mixing bowl, and an initial 5 ml aliquot of the linseed oil is added to the bowl. After the rise and subsequent fall in the torque value, the volume occupied by the powder decreases, permitting the addition of the rest of the powder. All the powder is then added and mixing is continued until torque equilibrium is achieved. If the volume occupied by the mix appears to be very small on visual examination of the bowl, an additional volume of the powder and oil may be added at this stage. Otherwise, titration with the oil is started. Once the amount of powder to be taken to ensure that the mixing bowl is loaded to at least 50% FBV is known, a second experiment may be carried out to accurately establish the point of CBVC. Thus, the amount can be assessed by a simple, initial trial and error procedure.

3.2.3 Mixing speed

Figures 5 (a) and (b) show the variation of torque as a function of the binder content in the mix, for three different rotational speeds of the mixing blades, using coarse silica as well as fine alumina powder. It is clear from the figures that the point of CBVC is not altered significantly by the speed of the blades for both the coarse as well as the fine powders. For speeds below 50 rev min^{-1}, however, the time for attaining equilibrium torque levels after each addition of oil becomes longer and longer, thereby increasing the time required for the experiment. A rotational speed of 50 rev min^{-1} is found to be optimal, as the experiment can be completed within about 30 min. Higher speeds can lead to high wear and hence are not particularly advantageous.

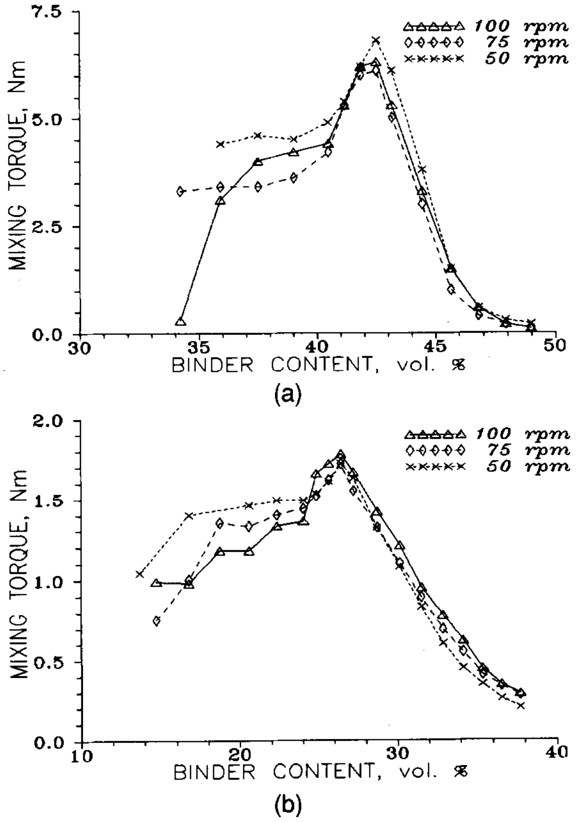


Fig. 5. Equilibrium mixing torque as a function of binder volume concentration for different speeds (rev min^{-1}) of the mixing blades: (a) 75 g fused silica and (b) 100 g alumina.

3.2.4 Mode of oil addition

Figures 6 (a)–(c) show the torque–time curves during mixing of fused silica powder, with linseed oil added in a stepwise manner as well as in a continuous fashion. Figure 7 shows the torque data from the above curves as a function of the binder content in the mix. In the case of stepwise addition of oil, the torque is allowed to stabilize before the addition of the next aliquot of oil. The shape of the torque–time curves appears distinctly different when the oil is added continuously, compare Figs 6 (b) and (c) with Fig. 6 (a). However, this apparent difference does not really manifest itself in Fig. 7, implying that the torque vs. binder content relationship remains essentially the same irrespective of the mode of addition, and that the point of CBVC can be identified quite unambiguously in all three cases. While conducting the experiment in a Brabender Rheometer, only torque–time curves are available, and therefore continuous addition of the oil at a rate of about 0·6 to 2·3 ml min^{-1} clearly offers a better alternative.

3.3 Applicability

The method has been applied to many ceramic and metal powders of different size, size distribution and shapes. Table 2 presents the particle characterization data for the different powders analysed, while the CBVC values are shown in Table 3. While the applicability of the method appears to be quite universal, special care is necessary while analysing submicrometre-sized powders. Fine and highly abrasive powders can easily penetrate the regions near the shaft of the mixing blades. Abnormal torque fluctuations signal such a possibility. The problem can be corrected normally by careful cleaning of the mixing chamber and the blades before fixing the mixing blades to the mixer.

3.4 Error analysis

The error in the method arises from the inaccurate

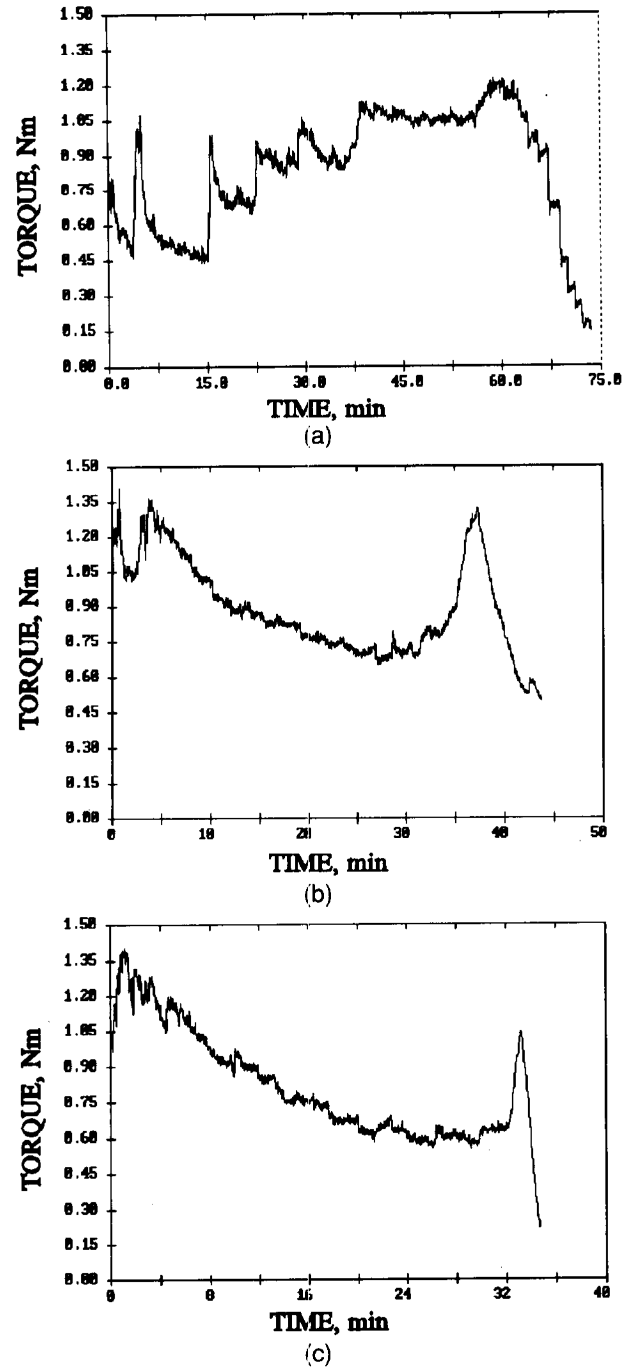


Fig. 6. Torque–time curves for 65 g fused silica at 50 rev min^{-1} using (a) stepwise addition of oil; (b) continuous oil addition at 0·6 ml min^{-1} and (c) continuous oil addition at 2·3 ml min^{-1}.

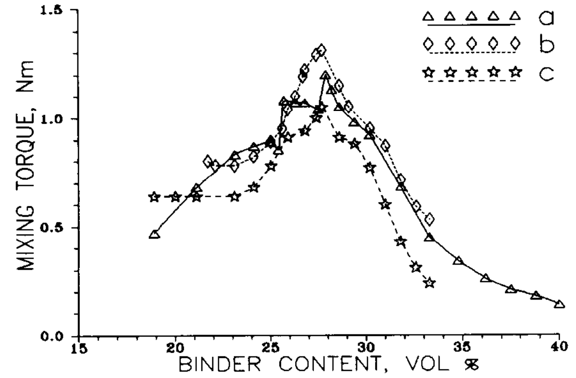


Fig. 7. Mixing torque vs. binder volume concentration graphs for the data from Fig. 6. Note the lack of any significant difference amongst the three curves in contrast to the sharpness of the end points in Fig. 6 for continuous oil addition expts.

recognition of the end point as well as from the statistical uncertainty. The torque during mixing is not a very stable value, but keeps fluctuating around a mean, even when mixing equilibrium has been achieved. Because of this, if the changes in the torque values are not significant for small additions of oil very near the point of CBVC as seen in Fig. 8, the accurate identification of the end point becomes difficult. In such a case, there will be some uncertainty in volume of oil at the end point. Table 3 presents this error data for different powders.

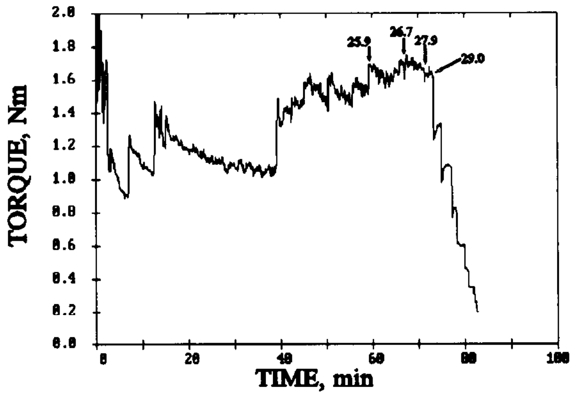


Fig. 8. Torque–time curves for a 75 g fused silica sample at 50 rev min^{-1} showing the fluctuations in the torque near the point of CBVC which leads to the uncertainty in the value of CBVC.

Table 2. Powder particle characteristics

Powder	*Particle Characteristics* Average size (μm)	Shape	Size distribution	Source
Fused silica	35	Angular	Multimodal	Remet Corp., USA
Alumina	2	Irregular	Monomodal	Alcon, USA
Coarse SiC	58	Irregular	Bimodal	Carborundam, India
Fine SiC	16	Irregular	Bimodal	Carborundam, India
Zirconia	2	Irregular	Monomodal	IRE, India
Fine silica	< 1	–	Monomodal	Elekem, Norway
Fine zircon	6	Angular	Multimodal	Casting Supplies, USA
Coarse zircon	18	Irregular	Multimodal	IRE, India
Coarse nickel	150	Spherical	Monomodal	DMRL, India
Fine nickel	12	Irregular	Multimodal	DMRL, India
Coarse alumina	75	Irregular	Monomodal	Alcon, USA

Table 3. Error in end point recognition for different powders

Powder	*Powder Weight (g)*	*Oil volume at CBVC (cm^3)*	*CBVC (vol%)*	*Max. Relative error in CBVC (%)*
Fused silica	65	12·0	28·7	±2·7
Alumina	99	18·5	42·5	±1·6
Coarse SiC	155	26·0	35·0	±2·5
Fine SiC	110	27·0	44·0	±4·2
Zirconia	145	23·5	48·9	±3·3
Fine silica	25	15·5	57·4	±4·1
Fine zircon	150	9·8	23·4	±5·9
Coarse zircon	117	15·8	38·6	±4·9
Coarse nickel	250	20·5	42·3	±4·2
Fine nickel	160	25·2	58·4	±2·1
Coarse alumina	99	32·5	56·0	±2·3

The statistical uncertainty in the value of CBVC was obtained by repeated analysis of FSU powder. The mean value of CBVC from 11 determinations was 27·3% with a relative standard deviation of 3·5%. It is thus clear that CBVC can be determined by the proposed torque rheometric method with good reproducibility.

4 Conclusions

Critical binder volume concentration (CBVC) represents an excellent estimate of the minimum amount of a binder liquid necessary to impart fluidity to a powder mass, and its complement, the critical particle volume concentration (CPVC), denotes the maximum attainable packing density for a given powder. The method described for the estimation of CBVC is simple, fast, accurate and universally applicable to ceramic or metal powders of different sizes and shapes. Various parameters affecting the determination have been studied. The method has been applied to a variety of powder samples.

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