

Statistical Characterization of Particulate Properties of Some Industrial Powders

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Abstract. In this investigation, the statistical optic characterization approach is covered for four industrial powders, namely; gelatin, iron, tin and sodium chloride. For all of these powders few size fractions from sieve analysis are optically analyzed and the particles shape and size distributions are obtained. A discussion of the effect of these primary properties statistical distributions on some of the physical parameters (i.e. the secondary properties) including friction, flow and density is presented. A standard sample size determination technique for proper number of experimental data (i.e. particles) to obtain reliable and accurate results is utilized. Several standard statistical probability density function are attempted in order to obtain the best fit of the experimental statistical diameter histograms of particles. The Gamma probability function is found to be a suitable statistical model to fit the experimental particle diameter histograms of the four powders. Finally, a non-linear correlation is displayed between the fitted distribution function parameters and the measured secondary powder parameters.

Introduction

Many industries at some stage require to produce, handle, characterize, analyze and control matter in the particulate form. Thus, to insure the success, efficiency and profitability of these industries, an extensive knowledge of the characteristics of the particulate materials is essential. Furthermore, the importance of airborne particles to humanity must be obvious to most persons. It is clear that we all live in a great aerosol system, our atmosphere, and the particles it contains have vast influences on our climate, health, work, etc. Much of our technology involves the production, use and often the elimination of airborne particles [1]. Thus, studies of airborne particles are an important aspect of industrial hygiene, weather modification, and studies of the natural atmosphere. Often the most important part of any aerosol investigation is measuring the suspended

particles, which includes the determination of particles size distributions, concentrations, and shapes. Also, fluid containing micrometer and submicrometer sized particles have been found to be responsible for yield losses in the manufacture of sophisticated and sensitive electronic components and other materials. The detection of these particles is dependent on the physical and chemical properties of both the particles and the fluids that contain them. This is particularly true in the particulate industrial pollution problem, where the particle detection, collection and characterization as well as particle control and removal techniques and transport are critical. Thus, to achieve efficient contamination control and have an insight to particulate industrial pollution, it is essential to be able to measure and characterize the contamination particles [2,3].

Particle technology is a highly complex subject, where fundamental scientific quantitative formulations and techniques are difficult to achieve. However, from the vast amount of research available in the area of powder characterization, it is possible to group the main parameters used into two categories: (i) The primary characterization parameters such as particle size and shape; (ii) The secondary characterization parameters such as density, flow, friction, etc. Many properties of particulate system, however, can be synthesized from these basic particle parameters [4-7].

Generally, powders are known to come into different shapes and sizes, which affect their behavior. Much effort has been expended over the last few decades to identify the significant parameters of the powder which affect its properties and characterization. Many particle characterization techniques are available and widely used [4-7]. Extensive data have been obtained from tests performed on regular shapes of particles (e.g. spheres, cylinders, etc.) employing some statistical treatments. But the statistical optic characterization (i.e. size and shape) of some manufacturing powders is still obscure. However, very few scant attempts are made to develop statistical methods and statistical probability density functions to explain the behavior of powders with irregular particles. Amongst these are that of Lewis and Goldman [8,9], Stockham [10], Itoh *et al.* [11], Barret *et al.* [12], Okaya and Toshio [13], and recently by Es-Saheb and Cogun [7]. But, when applying these techniques to the actual real powders, there has been varying degrees of discrepancy among the results of the different investigators, particularly when considering irregular shapes of particles.

Thus, in this investigation these issues are addressed and the statistical optic characterization approach is utilized for four different industrial powders, namely, gelatin, iron, tin and sodium chloride. For all of these powders size fractions from sieve analysis are optically analyzed and the particles size and shape distributions are obtained. A discussion of the effect of these primary properties statistical distributions on some of the secondary physical parameters including friction, density and flow properties is presented. Sample size determination technique used to calculate the required number of particles to be observed to obtain reliable and accurate results for the primary characterization of the powder is presented. Several statistical probability density (i.e.

distribution) functions are tried to obtain the best fit of the experimental statistical diameter histograms of particles. An attempt is made to correlate the fitted distribution function parameters and the measured secondary powder characteristics.

Materials and Experimental Methods

The four different powders used in this investigation are: Iron powder produced from cast iron by fine filing (Produced by Hopkin and Williams Ltd., England); Pure tin powder (produced by Seelze-Hannover, Germany); Gelatin gold normal powder (produced by Riedel, Germany) of AG.D3016(18807) type and; Sodium Chloride extra pure in its normal laboratory form (produced by Merk, Germany).

As the case with all the powders, we started the characterization with the standard sieve analysis test, where the particle sizes and size distribution for all four powders investigated are obtained. Then, to complete the characterization of these powders, including the shapes and shape frequency distributions of the particles, optic-microscopic investigation is employed. Several glass slides of each powder are prepared and examined under the microscope (Vanox type, Olympus Ltd.). Photographs are taken for hundreds of particles, (about 1000 particles) of each powder for later measurements and processing. The details of these tests and the measurement techniques used together with the statistical manipulations, sample size determination, statistical modeling of particle characteristics and the discussion of the results are presented in the following sections.

Particle Size

The term "particle" can be defined as any object having precise physical boundaries in all directions [1]. One of the most common definitions of particle "size" is diameter. But, the term diameter (or radius) is unambiguous only for spheres. When all the particles in a system have the same shape (such as cubic in the case of sodium chloride), then the problem of defining diameters in terms of the dimensions of the particles is relatively easy. However, when the particles vary markedly in shape (as the case with the rest of the powders under investigation), diameter definition is much more difficult.

Diameters of irregular particles can be defined in terms of the geometry of the individual particles or in terms of their physical properties. Diameters of the former type are often described as statistical because they have much meaning only for averages of the measurements of a large number of irregular particles. Sieving, as a method of size determination, which is employed in this investigation, can be considered to yield statistical diameters, since the results depend directly on the dimensions of the particles and are averaged by the method for a large number of particles. Typical particle size distribution for gelatin, tin, iron and sodium chloride are shown in Fig. 1.

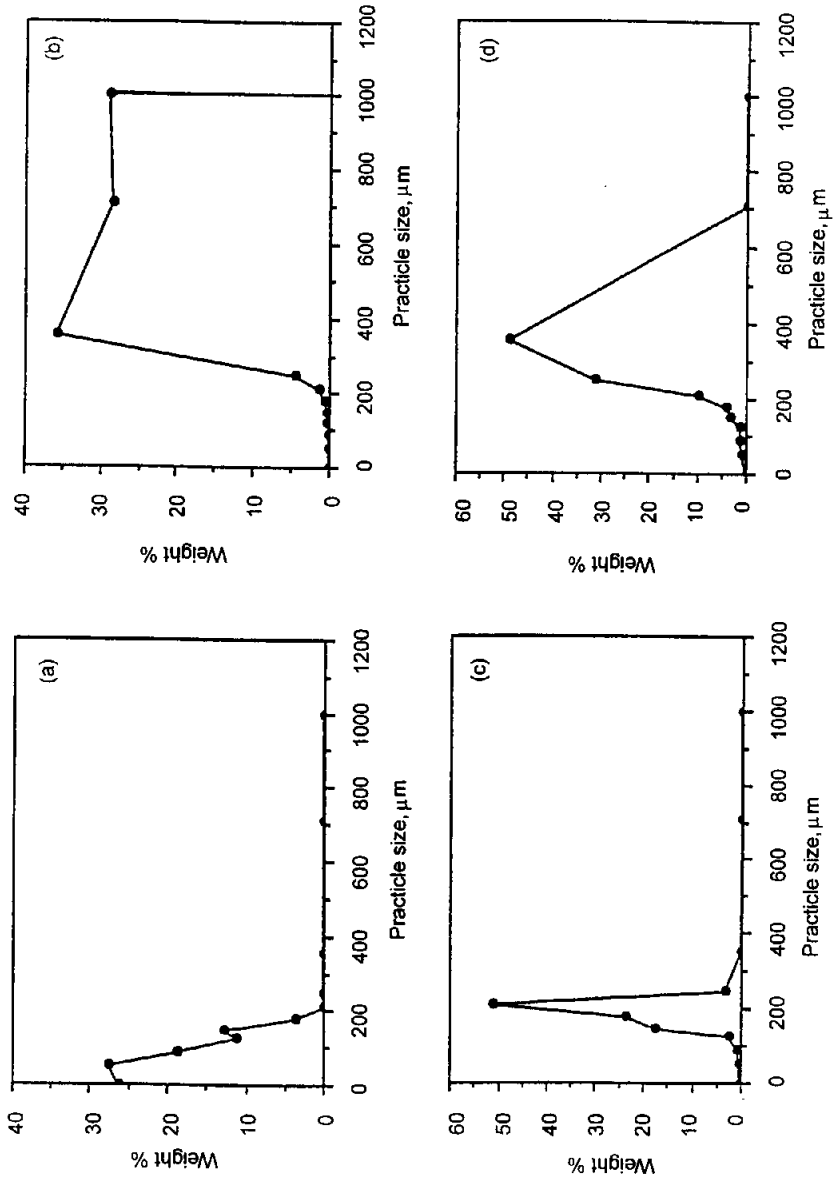


Fig. 1. Particle size distribution obtained from the sieve analysis tests for powders: (a) iron, (b) gelatin, (c) tin and (d) sodium chloride.

Perhaps the most widely used statistical diameter is the diameter of a circle (d) whose area is the same as that of the area of the particle (A) projected onto a surface. This technique is utilized in this study where, $d = \sqrt{4A / \pi}$. It is originally used for particle measurement by optical microscopy, which employed in this study. In this text, the

statistical diameter of the particle ($d = \sqrt{4A / \pi}$) will be shortly referred to as "statistical diameter". Figure 2 shows typical statistical diameter histograms of this type (i.e. the statistical diameter vs frequency) for iron, gelatin, tin and sodium chloride powders, respectively. This definition, however, is easy to apply when the particles are regular (i.e. individual) in shape, but it is almost impossible to apply to certain aggregates and particles collected on hard surfaces, for example, by impaction during sampling for pollution assessment.

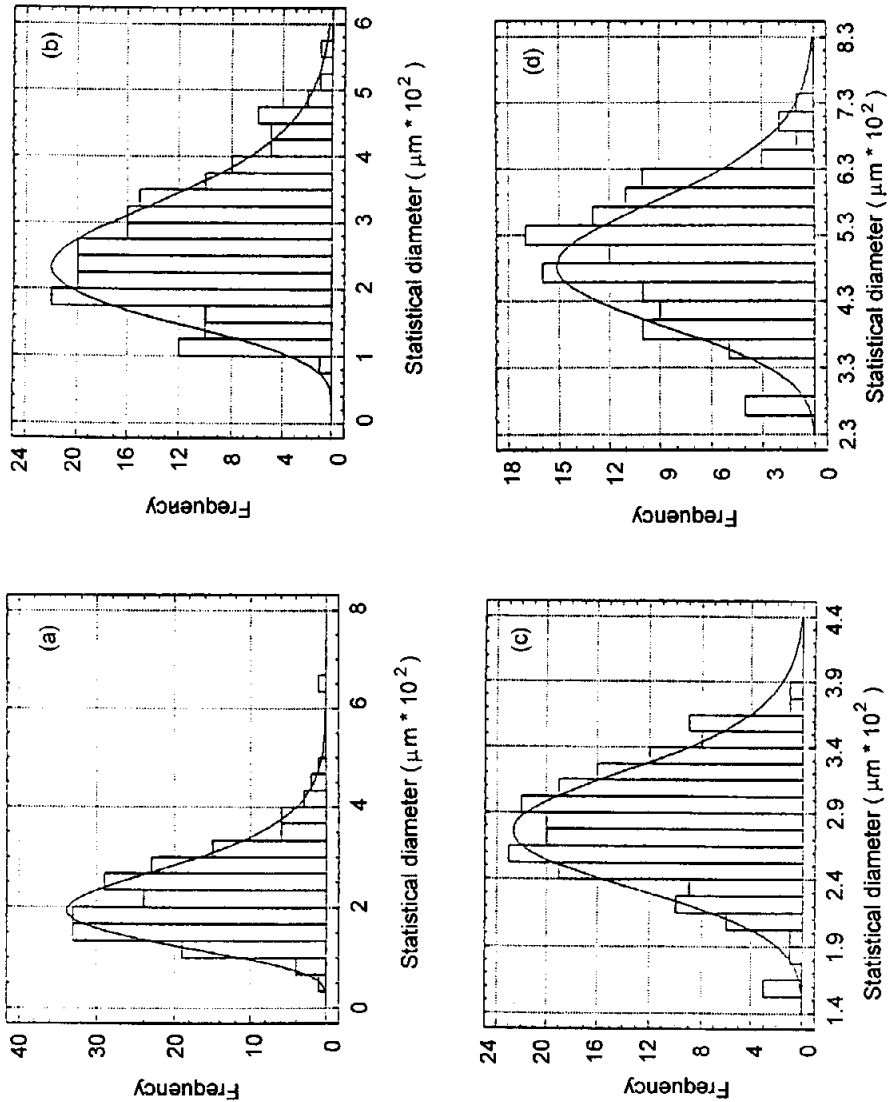


Fig. 2. Statistical diameter histograms of the particles, (a) iron, (b) gelatin, (c) tin and (d) sodium chloride.

Other definitions of diameter are the arithmetical average of the dimensions, length (l), breadth (b), and the thickness (t) of the particle; ($d = (l+b+t)/3$), or the length of the side of a cube of equal volume; ($l = \sqrt[3]{lbt}$). These definitions assume that all particle dimensions can be measured. This, however, is not true in our case as the images (photos) obtained for all particles are two-dimensional. Furthermore, other diameters are also proposed, among these is the Martin's diameter and Feret diameter [14]. Finally, elongated particles such as fibers may be described merely by length, by thickness, or by both. Diameters defined in terms of physical properties, which are not employed in this study, are determined by measuring properties such as light scattering, sedimentation rates or rates of Brownian diffusion [1].

Particle Shape

Defining particle shape is often even more difficult than defining particle size. In spite of this difficulty, many researchers proposed different parameters or definitions for characterizing the particle shape. Most of these attempts, however, are based on (i) measuring the dimensions of individual particles [15], and (ii) estimating the mean geometric properties from two different properties of the particle, which are influenced to different extents by the shape of the particles [4].

Heywood, [15] proposed that the particle be assumed to be resting on a plane in the position of greatest stability. He, also, defined flakiness, F , as b/t (where b and t are the breadth and thickness of the particle), and elongation, E , as l/b (where l is the length of the particle). Meanwhile, Hausner, [4] has proposed a method for characterizing particle shape based on the dimensions of a rectangle drawn around the projection of the particle. He proposed the use of three dimensionless ratios, called the elongation factor, the bulkiness factor, and the surface factor, to characterize the shape of the particle regardless of its size. This, of course, is a two-dimensional treatment. This approach however is adopted for characterizing our powders in this investigation. Figure 3 shows a schematic presentation for characterization of particle shape using the method of Hausner, [4]. It is suggested that the rectangle of minimum area drawn around the particle projection be used. The elongation of the particle is given by,

$$x = a' / b' \quad (1)$$

where x is the elongation factor, and a' and b' are the side lengths of the rectangle, a' being the longer. Meanwhile, the bulkiness factor y is given by,

$$y = A / (a'b) \quad (2)$$

where A is the projected area of the particle. Hausner [4], also defines a dimensionless surface factor, z , which is the ratio of the square of the circumference, (C), of the

projected area A of the particle to the square of the circumference of the sphere of cross-sectional area A ,

$$z = C^2 / 4\pi A. \quad (3)$$

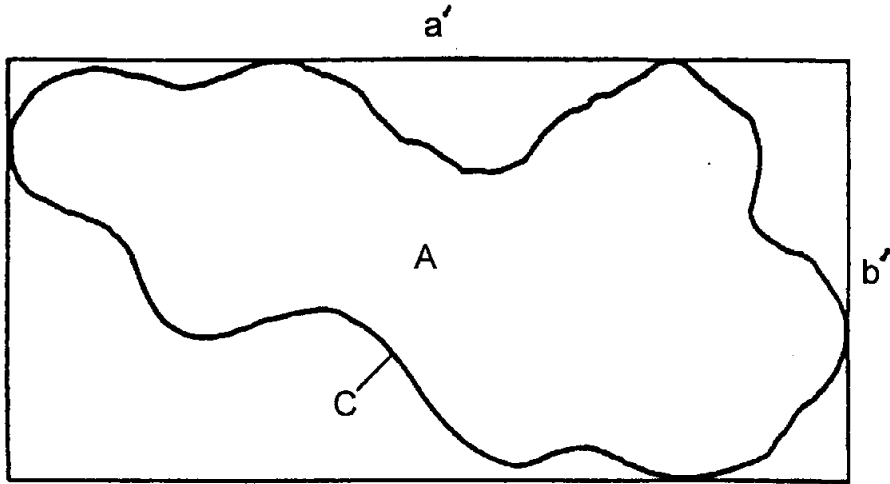


Fig. 3. Characterization of particle shape by Hausner [4].

The more the particle deviates from spherical shape, the larger will be the value of z . Figures 4-6 display the calculated values (histograms) of these factors (i.e. the elongation, bulkiness, and surface factors) for the investigated powders; gelatin, iron, tin and sodium chloride, respectively.

However, the approach to estimate particle shape based on measuring average properties of particles has been discussed in great details by Herdan, [16] and involves the use of the shape factors. The shape factors used are the surface (α_s) and volume shape factors (α_v), which are defined as $\alpha_s = S/d^2$ and $\alpha_v = V/d^3$, respectively, where S is the surface area, V is the volume, and d is the diameter of the particle [1, 15]. Other parameters or terms, such like rugosity factor, to describe the particle surface, are also used [17]. Furthermore, we can generalize the principle of the use of shape factors by stating that the ratios between diameters obtained by different particle sizing methods are related to average particle shapes and are essentially constant for a given type of particles. The proportionality factors are the shape factors usually defined as the proportionality constants between mean diameters determined microscopically for individual particles and those determined for the entire particles.

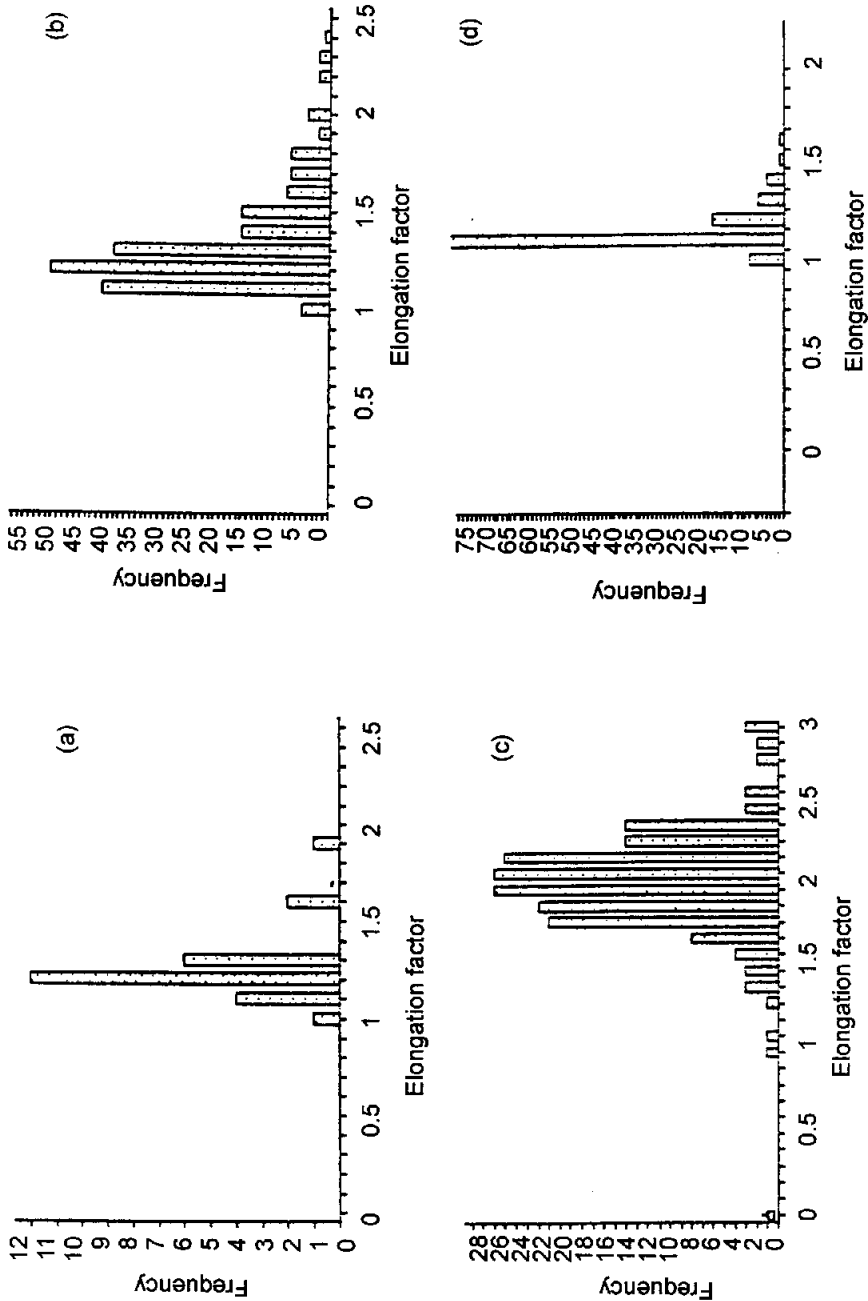


Fig. 4. The elongation factor histograms for powders, (a) iron, (b) gelatin, (c) tin and (d) sodium chloride.

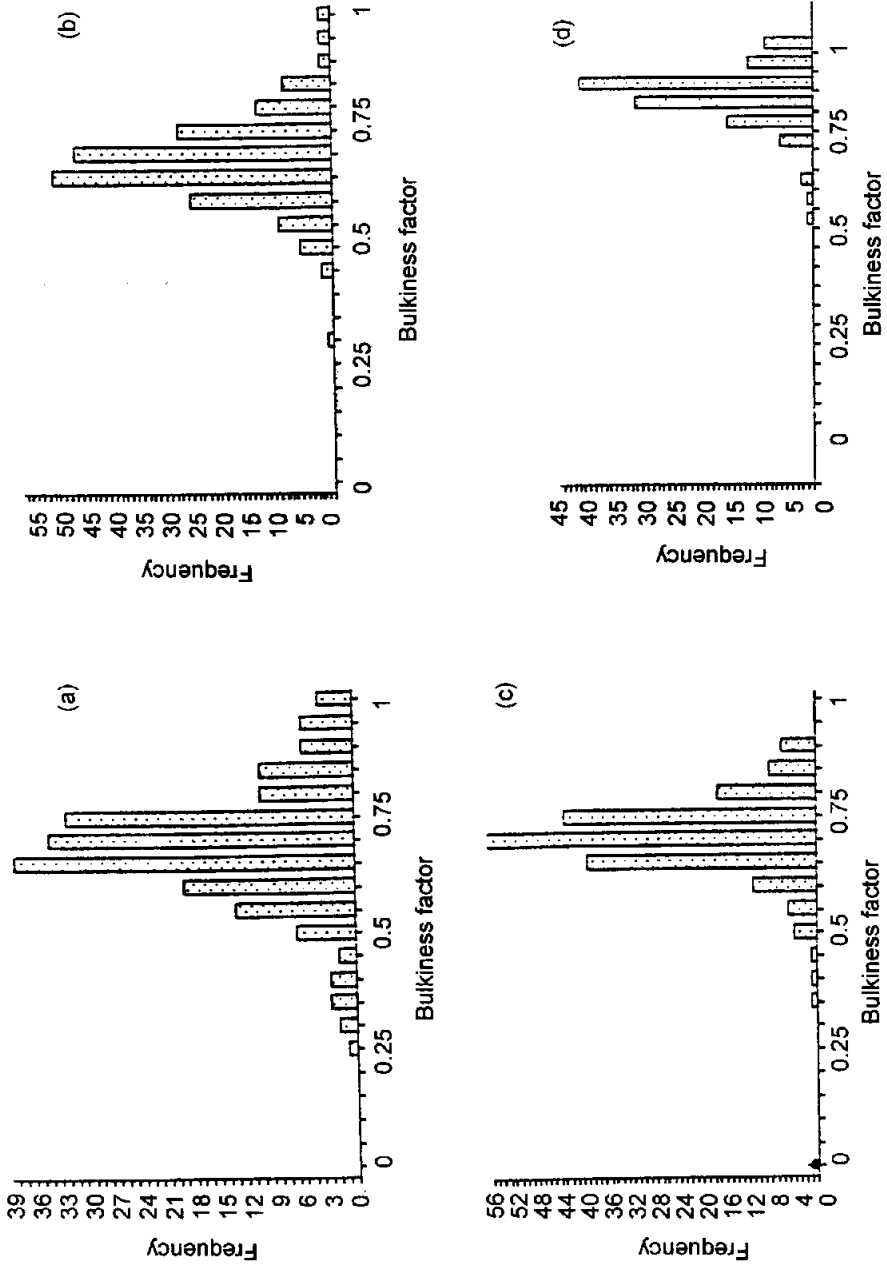


Fig. 5. The bulkiness factor histograms for powders, (a) iron, (b) gelatin, (c) tin and (d) sodium chloride.

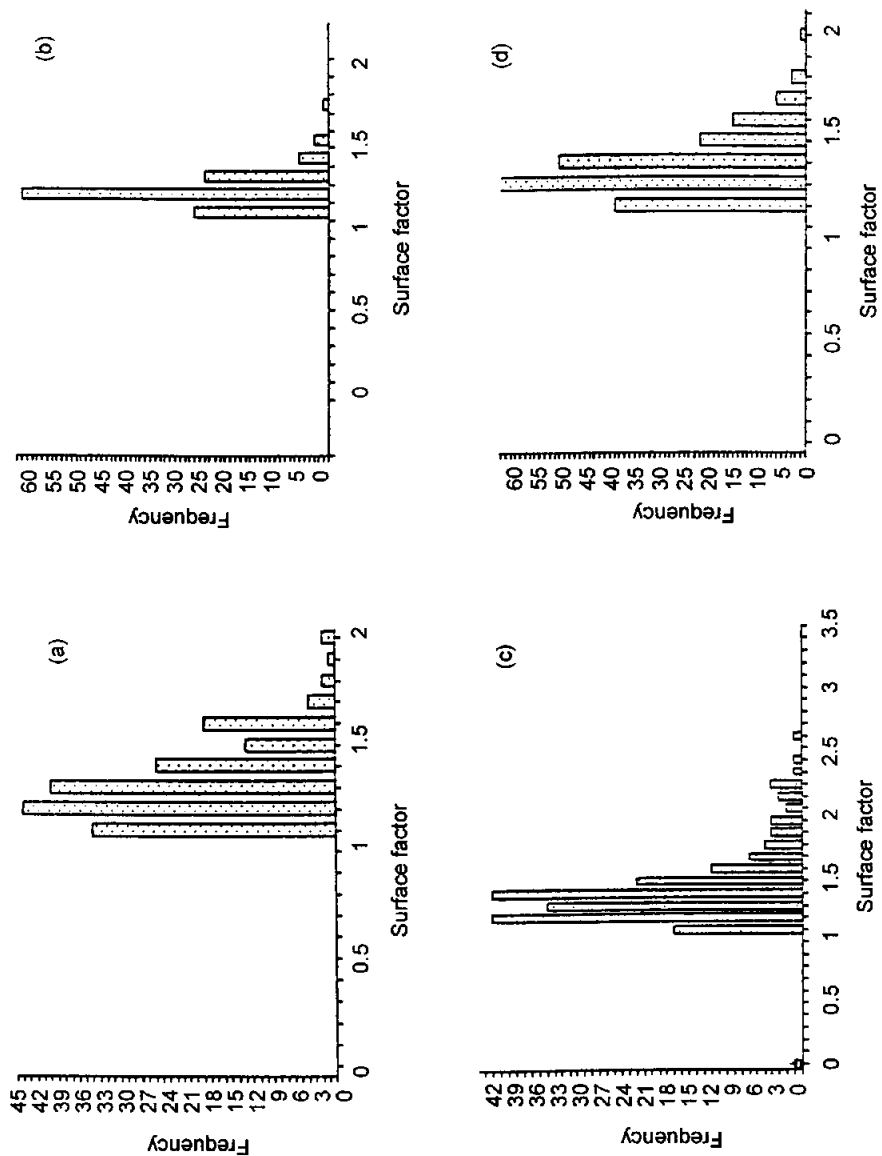


Fig. 6. The surface factor histograms for powders, (a) iron, (b) gelatin, (c) tin and (d) sodium chloride.

As stated above, though the average diameter is considered to be the most useful dimension, widely accepted to reflect the particles size, (Table 1), it is not sufficient to give all the information needed about the particles shape. Thus, in principal, the use of the average shape factors, such like α_1 and α_2 , to provide some description of the particles shape can be employed. But in order to have any specific particle shape information,

other shape factors are required; such like the elongation, bulkiness and surface factors [4], adopted in this investigation. Figs. 4-6 displayed the statistical distributions of these factors for all powders studied. However, the average values of the elongation factor (x), see Fig. 4, are found to be 2.19, 1.29, 1.07 and 1.25 for tin, gelatin, sodium chloride and iron powders respectively. Also, the average values of the bulkiness factor (y) for the powders in the same order are, 0.68, 0.66, 0.84 and 0.67, see Fig. 5. Meanwhile, the powders average values of the surface factor (z), in the same respected order are 1.39, 1.24, 1.17 and 1.26, see Fig. 6. From the values of these factors it is clear that all powders particles studied are of irregular shapes. However, for sodium chloride powder it is noticed that, the elongation factor is close to 1.0 indicating that the particles are not angular in shape, also, the surface factor is close to 1.0 which is an indication of regular or close to spherical shape, meanwhile, the bulkiness factor is found to be close to 1.0 suggesting that the particles shape is near to cubic. Also, for the tin powder it is observed that, the elongation factor and bulkiness factor are far from 1.0 while the surface factor is close to 1.0, this indicates that the particles shape is near to the angular shape. But, as far as the iron and gelatin powders are concerned, it is found that the shape factors for both powders are almost the same and the values of the three shape factors considered are far from 1.0, concluding that the particles shapes are irregular in both cases. These findings are in good agreement with the visual inspection of the particles micrographs obtained for all powders [24]. This, however, shows the importance and the need for the real powder particles photographs in order to have a complete particle shape description. For more details on these issues, including particles pictures, statistical treatments, measurements, etc.... see ref. [24].

Particle Statistics

It is clear from the above results that powders characterization including the particle sizes, shapes and their distributions are of statistical nature. Since the powders in general never consist of particles that are exactly the same size and shape, analysis and modelling of such data by statistical methods are very enlightening. The contribution of statistical analysis and modelling of the particles to the characterization of the powders can be summarized as follows: If sufficient particle information (diameter, bulkiness factor, elongation factor, shape factor, etc.) are collected for a certain powder, it would be possible to model the behavior of this powder by using statistical analysis. This in turn, would provide a powerful tool for the prediction of the behavior of other powders {characterization (i.e. shape and size characteristics) and processing (i.e. compaction, flow through filters, etc.)} which could be described with the same statistical model and model parameters without any further experimentation. Some statistical aspects of particle characterization has been established in the literature [3,7,8,14,16-18] but however, still more work and research is needed in this sophisticated field.

In the process of statistical analysis and modelling of the powder properties, the first step is the determination of the required number of particles to be measured (i.e. sample size) which would be large enough to reflect the characteristics of powder (i.e. population). This is presented next.

In all published research work on the determination of statistical particle diameter, extensive experimental efforts have been spent to obtain reliable results. In these studies, only the consistency of experimental results (average statistical diameter) obtained from the same powder is considered as the criterion for the reliability of results. In the literature, no published information is found on sample size determination for diameter measurements of particles. Thus, prediction of sample size for reliable experimental data would be very useful, particularly in cases where large experimental efforts are required.

The principles of sample size determination is discussed in many statistics books [19, 20] and the following well known formula is used to determine sample size:

$$n = (Z_{\alpha/2}s/E)^2 \quad (4)$$

where n is the sample size, $Z_{\alpha/2}$ is the upper critical value from normal tables with a confidence of $(1-\alpha)$, s is the known population standard deviation and E is the maximum acceptable error. Commonly, unknowns s and E are estimated by using some preliminary (pilot) experiments or by using past data. In this study, for each powder 200 particles are considered as infinite (parent) population by using the authors past experience. The following conditions are applied in order to determine the sample sizes for all four powders, each containing 200 data (diameter) points; (1) 5% maximum acceptable error of population mean (i.e. mean of statistical diameters), (2) 95% confidence, (3) mean and standard deviation of population (diameters) estimated from 200 samples (see Table I).

As shown in Table I, for the four different powders, different sample size values ranging from 52 to 183 are found by using the above mentioned method. The results clearly indicate that it is not possible to find a standard sample size for statistical diameter measurements for powder characterization. Statistically, it is shown that 200 particles (measurements) for each powder are large enough random sample to reflect the characteristics of the population (powder).

Table 1. Sample size determination for particle diameters for the four powders

Powders	No. of experimental data	Average particle diameter [μm]	Particle standard deviations (S)	Sample size estimation
Tin	200	2.77	0.424	52
Gelatin	200	2.7	0.924	183
Sodium chloride	200	5.01	0.914	52
Iron	200	2.24	0.735	152

Statistical Modeling of Particle Size Variation

The frequency histograms for the statistical particle diameter for the four powders are given in Fig. 2. Careful visual analysis of the frequency distributions revealed the fact that Gamma, Erlang, Lognormal, Normal, Triangular and Weibull distribution functions (models) could be possible probability distributions to describe the physical phenomena related to the random behavior of statistical diameters of a powder. Those models are assumed due to their wide variety of shapes, especially Gamma and Lognormal models, which has been used to model similar events in different systems and processes [21].

In order to assess the applicability of the above mentioned models in particle characterization, it is necessary to statistically test to see if models (probability distributions) with estimated parameters actually fits the experimentally observed (measured) particle diameter information. There are various tests to check the goodness of fit. One of the commonly used tests in statistics is the chi square goodness of fit test. This test is used in this study. The general procedure for using chi square test can be found in most of statistical analysis books [22, 23].

Models were tested for chi square goodness of fit test for each powder by using a standard statistical program package STATGRAPH. Table 2 includes both results of the χ^2 and significance levels of the χ^2 goodness of fit. In the table, the higher the χ^2 values and lower the significance levels indicate the better fit of the hypothesized model to the experimental particle size distribution. Also, in the table there are some exceptionally low values of significance levels indicating excellent fit of the hypothesized model to the experimental particle size distributions as in the fit of the triangular distribution to the experimental particle size distributions of tin, or the fit of the log normal distribution to the experimental particle size distributions of sodium chloride. However, considering the overall significance levels, the Gamma model seems to be the best statistical model to fit the experimental frequency distributions for all powders. Gamma probability function is always skewed to the left and can assume a wide variety of shapes. It is therefore one of the most appropriate statistical models in applied science work. The probability distribution function of the two-variable Gamma model is given by,

$$F_G(t, \beta, \alpha) = 1 / (\beta \Gamma(\alpha)) (t/\beta)^{\alpha-1} e^{-t/\beta} \quad 0 < t, \alpha, \beta \quad (5)$$

where, β is the scale parameter, α is the shape parameter, $\Gamma(\alpha)$ is the Gamma function and t is the particle diameter (random variable).

Table 2. Chi square test results and probability function parameters for the four powders

Powders		Probability function (model)					Weibull
		Gamma	Erlang	Log-normal	Normal	Tri-angular	
Tin	Chisquare value	10.64	10.6	13.08	3.18	16.36	9.37
	Sig-level	0.474	0.478	0.288	0.988	0.128	0.59
Gelatin	Chisquare value	9.23	10.31	17.07	23.19	22.98	16.83
	Sig-level	0.683	0.589	0.15	0.026	0.028	0.156
Sodium chloride	Chisquare value	8.18	8.16	10.84	5.34	6.66	5.3
	Sig-level	0.416	0.416	0.287	0.72	0.67	0.725
Iron	Chisquare value	5.73	5.85	6.12	21		16.595
	Sig-level	0.68	0.67	0.63	0.0072		0.056

The curves on the graphs in Fig. 2 show the fitted gamma distribution function (with the model parameters) calculated by using the probability function given above (Eqn. 5). In Table 3, the calculated gamma probability function parameters by STATGRAPH package for each powder together with the measured physical properties of powders (i.e. flow time, angle of repose, apparent density and tap density) are given.

In Fig. 7, gamma distribution parameters as well as the physical properties of the four powders are shown. However, more detailed research work is necessary to establish well, the variations of statistical model parameters with physical properties of the powders and diameters of particles. This issue is addressed and discussed by Al-Matery, [24], where, more details on the materials, measurements techniques, statistical procedures, etc. can be found.

Table 3. Variation of gamma probability function parameters with the physical properties of the four powders

Powders	Gamma		Flow time (sec.)	Angle of Repose (deg)	Apperant density (g/cm ³)	Tap density (g/cm ³)
	Alpha(α)	Beta (β)				
Tin	40.53	14.33	34.66	19.6	4.12	4.2
Gelatin	7.76	2.91	-	27.54	-	1.07
Iron	7.17	3.20	38.16	24.72	3.02	3.46
Sodium Chloride	27.92	5.58	117.06	25.64	1.2	1.3

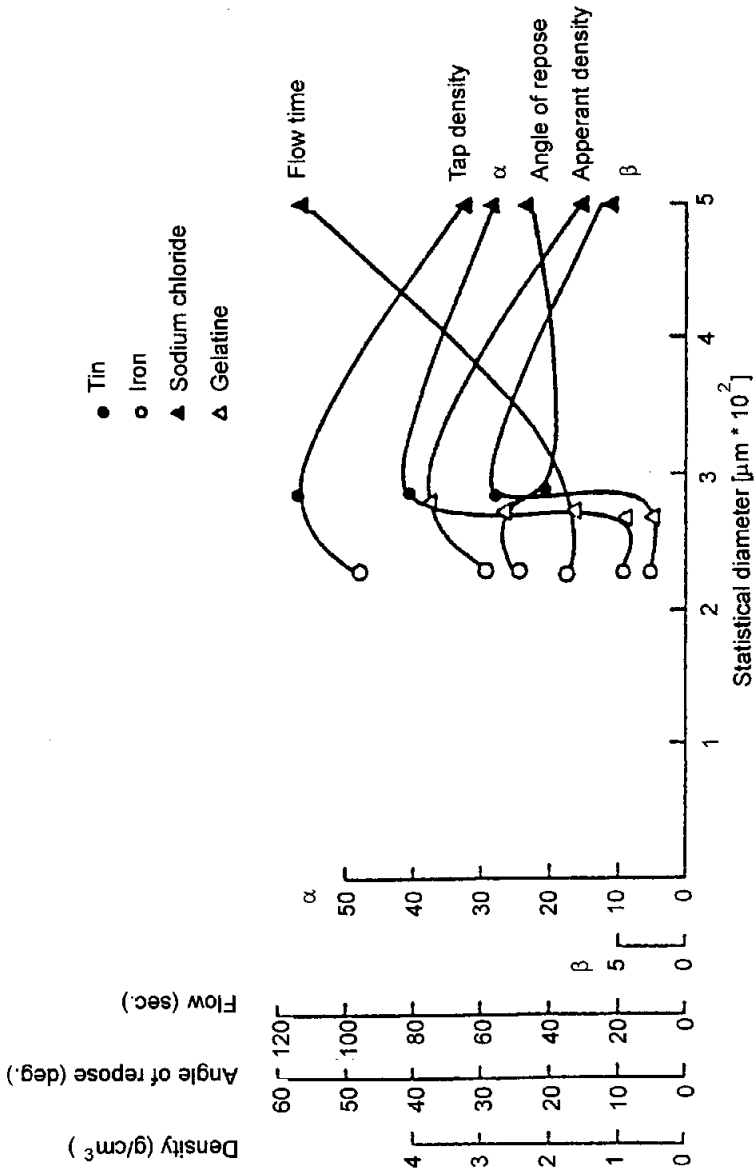


Fig. 7. Variation of gamma probability function parameters with physical properties of four powders.

Conclusion

The experimental and theoretical investigation of some statistical aspects of primary and secondary characteristics of four industrial powders, namely gelatin, iron, tin and sodium chloride is presented. Out of the many particle characterization techniques available and widely used, the statistical optic characterization, particularly for manufacturing powders, is found to be useful and most informative. As defining particle shape is more difficult than defining particle size. The particle size distributions (statistical diameters) of the four powders are found to be mostly left-skewed. Gamma, log-normal and Weibull distributions are found to be statistically acceptable models to reflect the behavior of the experimental statistical diameter distributions of the powders. However, regarding the chi-square and corresponding significance level values, the two-variable Gamma model shows the best fit. No unique (i.e. unified) standard sample size value for the statistical particle diameter, that can be used for the different powders, is found. However, 200 particles (i.e. statistical diameters) are found to be large enough random samples to reflect the behavior of the primary characteristics of each powder. Correlations between the physical properties of the four powders and both the mean statistical diameter and the estimated Gamma function parameters (scale and shape), are attempted and displayed nonlinear relations. Statistical modeling of powder primary characteristics would provide a powerful tool for the prediction of particle characteristics and physical properties of powders, without further experiments, over the investigated range. Finally, for efficient contamination control of industrial pollution, the measurement and characterization of the contamination particles are essential.

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توصيف إحصائي لخواص الحبيبات لبعض المساحيق الصناعية

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و** كلية الهندسة ، جامعة غازي ، أنقرة ، تركيا

(قدم للنشر في ١٠/١٠/١٩٩٩، وقبل للنشر في ٢٨/٠٣/٢٠٠٠)

ملخص البحث . تمت في هذه الدراسة تغطية طريقة التوصيف البصري الإحصائي لأربعة مساحيق صناعية هي : الجيلاتين ، الحديد ، القصدير وملح كلوريد الصوديوم . تم تعيين الخصائص الأساسية لكافة هذه المساحيق من قياسات حجم الحبيبات غير المنتظمة وشكلها وكذلك توزيعاتها بطريقة التحليل البصري لحبيبات فئات المساحيق المختلفة التي تم الحصول عليها بطريقة النخل ، وتحديدًا تلك التوزيعات الخاصة "بالقطر الإحصائي" ، ومعاملات أشكال الحبيبات مثل معامل الاستطالة ، والسطح والحجم . وكذلك تمت دراسة تأثير هذه التوزيعات الإحصائية على بعض العوامل الفيزيائية (أي الخصائص الثانوية) ومناقشتها ، التي تم قياسها أيضاً ، والمشملة على الاحتكاك وخواص الانسياب ، والكثافة (الظاهرية والنقر) . وللحصول على نتائج دقيقة وموثوقة فقد تم استخدام طريقة قياسية لحساب حجم العينة اللازمة من المسحوق وعدد الحبيبات التي يجب قياس الخصائص الأساسية لها . وقد جربت العديد من الدوال الإحصائية المعروفة على توزيعات الخصائص الأساسية التجريبية (أي الأقطار الإحصائية) لحبيبات كافة المساحيق بقصد الحصول على أفضل دالة تطابقها . وتبين أن دالة التوزيع

"جاما" هي أكثرها مطابقة للنتائج العملية (قياسات الأقطار الإحصائية) للمساحيق الأربعة . وأخيرا أظهرت العلاقة الناتجة بين دالة التوزيع جاما والخصائص الثانوية للمساحيق سلوكاً لا خطياً .