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GRAIN SIZE ANALYSIS TECHNIQUES IN AGRICULTURAL PRODUCTS

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Abstract: Reduction of grain size in processing of grain products for food and feed purposes is the most important and energy consuming process. The performance of animals consuming mixed feeds of different grain sizes may vary. Therefore, grinding efficiency is an important parameter and the use of grain size analysis in terms of machine technique; is one of the main parameters in the comparison of the performances of grinding purposes and grinding activities. In this study, the most commonly used grain size analysis techniques (sieve analysis, microscopic (image processing) analysis, laser diffraction analysis, electrical resistance analysis and sedimentation analysis) were tried to be given comparatively.
Keywords: grain size, analysis techniques, agricultural products, food technologies

1. INTRODUCTION

Investigations in food science and technology, whether by the food industry, governmental agencies, or universities, often require determination of food composition and characteristics. Making an appropriate choice of analytical technique for a specific application requires a good knowledge of the various techniques (Nielsen S S., 2017).

Grain processing, including cereal and pulse processing, is one of the oldest and most important of all food technologies and forms a large and important part of the food production chain. Grain pulses are grown widely throughout the world and their dietary and economic importance is globally appreciated and recognized (Tiwari et al, 2011).

Reducing the grain size in the processing of grain products for food and feed purposes is the most important and most energy-consuming process. The following two points need to be taken into account in terms of energy consumption;

- Grain size should be chosen appropriately for the purpose and avoid unnecessary excessively fine grinding.
- The mills to be used during milling should be sensitive to the selection of the construction and operating parameters.

Therefore, use of grain size analysis in terms of machine technique is one of the main parameters in the comparison of the performances of grinding purposes and grinding activities (having different constitutive properties and different operating parameters) in terms of grinding efficiency.

Over recent decades, various new methods for grain-size analysis have been developed (Stefano et al, 2010). The most commonly used grain size analysis techniques are; sieve analysis, microscopic (image processing) analysis, laser diffraction analysis, electrical resistance analysis and sedimentation analysis techniques.

Table 1. The sieve opening and mesh numbers of Tyler Ro Tap and USA

Elek açıklığı (µ)	Tyler Ro Tap (mesh/inch)	USA (mesh/inch)
3360	6	6
2380	8	8
1680	10	12
1191	14	16
841	20	20
594	28	30
420	35	40
297	48	50
212	65	70
150	100	100
103	150	140
73	200	200
53	270	270

2. MATERIAL AND METHOD

— Sieve Analysis

Sieve analysis is the oldest technique for measuring particle size distributions but is still a standard laboratory operation and extremely useful in practice (Bhandari et al., 2013). Sieve analysis test has been used as the main method to determine particle size distribution of granular materials including coarse materials for many decades (Kumara et al, 2012).

Elimination is the process of separating a solid grain mixture into components of different sizes using sieves. Size openings are based on a progressively decreasing standard series of elbow passes. The concept of "mesh number" is used when sieves used in screening are classified according to size. The mesh number indicates the number of holes per unit length (inch and mm) on one sieve. Tyler Ro Tap and USA standard sieve series are widely used. The sieve opening and mesh numbers of both sieve series are given in Table 1 (Baker and Herman, 1995). Also Figure 1 shows the sieve set and shake unit.

Figure 1 - Tyler Ro Tap sifter and shaker

— Microscopic Analysis

It is generally accepted as the reference method since the granules are the only method of direct observation and measurement. The method is based on grain counting at the microscope by dimensioning the granules with reference circles or scales (Saklara et al, 2000).

The microscope image is two-dimensional and the grain shape naturally deviates from spherical. In order to solve the problems that may arise due to these reasons and to ensure standard conditions, different diameters of the geometric meanings are defined. For example; Feret's diameter of a shape is a commonly used measure in shape analysis. Traditional methods for estimation of Feret's diameter are performed on binary images; one of these diameters is selected and counted. The number of areas to be scanned in the microscope and the number of grains to be counted in these areas are not given a definite number. However, it is recommended that at least 100 different fields and a minimum of 6 grain counts at a glance be recommended for a good analysis (Allen T., 1992).

Images are obtained with an optical microscope in the system. These images are animated by the CCD camera and transferred to the image monitor. It is then divided into 512x480 pixels, taken into memory and converted back into analogue. The dimensioning of the grains is done after distinguishing the grains touching each other, eliminating the grains cut at the edge of the image and filling the gaps in the grains. The image processing, analysis and measurement system is shown in Figure 2.

Figure 2. Image processing, analysis and measurement system

In calculating the grain size distribution, grain volume is assumed to be proportional to the weight. In other words, the intensities of all the particles in the sample are considered equal. Census results are converted to cumulative values in percentage by weight.

Microscopic grain size analysis can lead to long-term distraction. Therefore it is time consuming and tiring. Operator ability is more important in this method than others. It is also a difficulty in the method of placing the grains on the microscope slides homogeneously to prevent overtaking. The most advanced form of the microscope method is image analyzers. They work by scanning a photograph or a direct microscope image with a camera and electronically evaluating all information related to the particles.

— Laser Diffraction Analysis

Laser diffraction is one of the most popular methods of characterizing particles by measuring the light they scatter (Pan et al, 2017). The determination of grain size by laser diffraction analysis is based on the fact that the grains reflect the laser beam by breaking and the angle of refraction is inversely proportional to grain size. Also, many researches used laser diffraction particle-size analysis to rapidly determine size distributions of samples in various stages of disaggregation (Mason et al, 2011). In other words, large grains break the laser beam in small angle and small grains in big angle (Allen T., 1992). This is schematically illustrated in Figure 3.

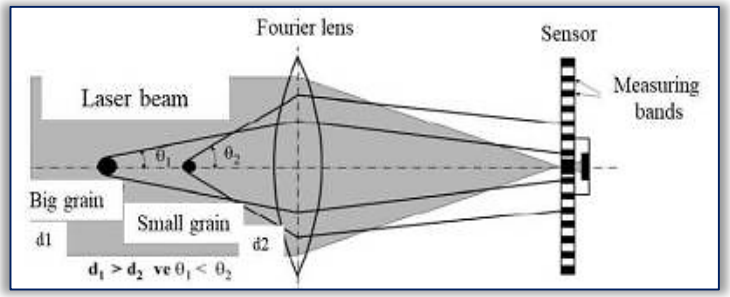

Figure 3 - Inversely proportional relationship between the size of the grains and the angle of refraction of the rays

This method, which is becoming increasingly preferred and prevalent in determining the dimensions of the particles ranging from 0.05 to 2000 µ, is based on the principle of pumping the particles in water and disintegrant mixed into a particular cell by means of a suitable device and forcing the laser beams to pass. As can be seen in Figure 4, laser diffraction devices usually come from three main parts: a laser unit, a sample preparation unit, and a computer (Özer and Orhan, 2007).

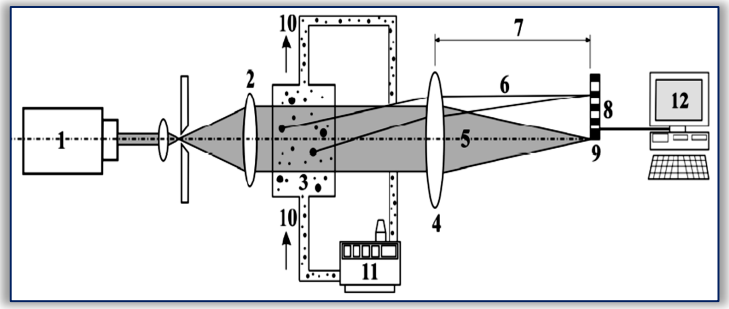

Figure 4 - General view of the laser diffraction device
1. Laser beam, 2. Beam expander, 3. Measuring cell, 4. Fourier lens 5. Beam bunch 6. Beam of the same size, 7. Focal length of the lens, 8. Multi-element detector, 9. Center detector, 10. Flow direction of the suspension, 11. Sample preparation unit, 12. Computer

By means of the unit-connected pump, it sends the particles in the form of a mixture of water and disintegrant into a special cell. The particles, which are pumped continuously into this cell, pass through the front of the laser beam several times. This cell was placed in front of the laser beam and exactly in the center. At this time, due to the lens (Fourier transform lens) located in front of the sample cell and the measurement band (data collecting detector) located behind it, the reflected beams are collected by being broken from the granules and continuously evaluated. When the rays reflected by the particles are reflected on the measurement band by the computer running simultaneously, the average volumes are calculated and the particle size is determined

One of the most important features of the laser diffraction method is to calculate the particle size distribution based on the volume of the particles. With this feature, it differs from sieve analysis, hydrometer and pipette methods based on the weight of the grains. Another feature is the use of equivalent sphere theory. In other words, it calculates the diameter of the grain in equal volume with the grain (Konert and Vandenberghe, 1997).

No matter how complicated and irregularly shaped the measured grains are, the volumes of the grains are calculated by means of the detector and the data collector, and this volume is presented in the form of the grain diameter by evaluating the equivalent round diameter. The most important advantages of this technique are; the weight of the granules used in the measurement and the specific mass are not needed. Because of the fact that the weight of the sample used in the experiment is avoided during the determination of the weight (Rawle A., 1995).

Laser diffraction technique has become very popular in recent years. The main reasons for this are; short analysis time, operator independence, repeatability, ease of use, applicable to almost all kinds of samples.

— **Electrical Resistance Analysis**

Electrical resistance analysis is an analysis of the relationship between the resistance of the granules to electrical current and their volume. The sample in an electrolyte is vacuumed into a glass tube with a circular opening at the bottom. The diameter of this opening may vary depending on the sample to be analyzed. Electric current is generated by the potential difference applied between the electrodes. As the grains pass openly, they show resistance to this electric current in proportion to their volume. This resistance difference determines the grain size. The electrical resistance diagram is given in Figure 5.

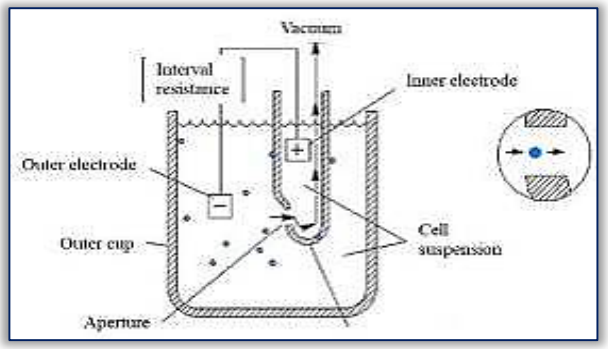

Figure 5 - Electrical resistance working diagram

The main discussed aspects of the method are incorrect transition and end point determination. In theory, it is assumed that the granules passed one by one through the tube opening. In practice, however, granules can be passed over and over again in the form of a binary, a triple, or a cluster, and erroneous readings can be made. To prevent this, the raw data obtained at the end of the analysis are corrected by the probability equations. In the equations developed for this purpose, the equality which is proposed by Coulter firm and which is 10% of the possible faulty transition ratio is accepted as general.

Another controversial endpoint concept is that the method is limited to temperature changes and electronic noise at the bottom of the measurement. Not all particles forming total volume can be counted by this method. The practical bottom dimension is 1 µm. The number or amount of grains under the lowest neck can be found using various interpolation techniques. The main idea in these techniques is based on the fact that the uncountable amount is subtracted from 100 (Allen T., 1992).

The most important advantage of the method is that the diameter found is the equivalent volume diameter. The effect of the particle shape on the measurement has been removed. Because the dimension statement is based on a geometric and physically strong point of view. In addition, providing a high resolution that is not available in other techniques is another important advantage (Hildebrve and Row, 1995).

— **Sedimentation Method**

There are numerous models of sedimentation in fine particle suspensions, derived from or validated with physical measurements (Benn at all, 2018). The sedimentation process is generally based on the principle that solid particles are precipitated in a liquid or gaseous under the influence of gravity. It is applied to determine the size of small grains (<0.075 mm) that are too small to be identified by the screen and the percentages within the total mass. For this, a sample with an approximate weight of 50 g is sieved with a 0.075 mm sieve. This sample is poured into a sedimentation cylinder made of glass with a diameter of usually 6.5 cm and a height of 45-50 cm and water is added to complete the volume to 1000 cm3. The precipitation cylinder is then agitated to bring the grain-water mixture to the same density throughout the cylinder at each point. The cylinder is then placed on a flat surface, allowing the particles to settle (Liu and Evett, 1997). Precipitation cylinder is shown in Figure 6.

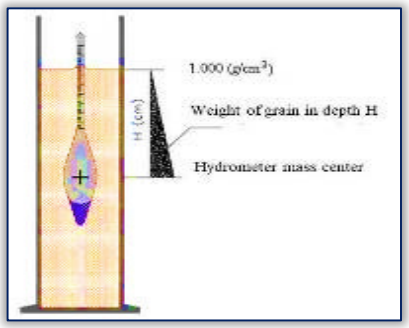

Figure 6 - Hydrometer measurement technique

This idea, which is the basis of the sedimentation analysis, was investigated by Stokes (1891) and mathematically stated that there is a relationship between grain diameters and sedimentation rates. This expression, which emerges with some acceptance, is called the "Stokes Law" in the literature (Rhodes M., 1998). If we are going to examine Stokes' law in more detail, which only examines the collapse of a single sphere in water;

A sphere with a diameter D is exposed to three different forces in a liquid with a viscosity η falling downward with a U limit speed (Bardet J.P., 1997). These are:

$$\text{The weight of sphere} = \frac{1}{6}\pi D^3 \gamma_s \ (+) \tag{1}$$

$$\text{Drift resistance} \quad = 3\pi\eta U D \ (-) \tag{2}$$

$$\text{Water buoyancy} = \frac{1}{6}\pi D^3 \gamma_w \ (-) \tag{3}$$

The sphere falling downwards with the force of weight, drift resistance and buoyancy forces of water are counterproductive. For this reason, we can assume that the first (+) and the second (-) are marked as shown above. After a certain period of time, the sphere reaches a constant speed due to these two opposing forces which counteract the falling sphere by accelerating downwards, which means that the forces are in balance. We can show this with the following equation:

$$\frac{1}{6}\pi D^3 \gamma_s - \frac{1}{6}\pi D^3 \gamma_w - 3\pi\eta U D = 0 \tag{4}$$

By organizing this equation, we can express the speed of attraction as a function of the height of the diameter:

$$U = \frac{1}{18}\frac{\gamma_s - \gamma_w}{\eta} D^2 \tag{5}$$

equality emerges.

However, it should be known that this is only true for laminar flow conditions where the Reynolds number is less than 1 (Re <1). Since it is not possible to measure the velocity of the falling particles in the precipitation cylinder, if we want to arrange the above equation:

$$H = \frac{1}{18}\frac{\gamma_s - \gamma_w}{\eta} D^2 t \tag{6}$$

equality occurs.

However, since the parameter we want to measure in the laboratory environment is not the path or precipitation distance of the grains, but the diameter of the grains, we have equally obtained the fundamental equality we use to measure the diameters of the grains by repeating D by pulling it back [11].

$$D = \sqrt{\frac{18\eta H}{(\gamma_s - \gamma_w)t}} \tag{7}$$

where: D = diameter of measured grains (mm), H = water viscosity, γs = unit volume weight of grains (g / cm3), γw = water unit volume weight (g / cm3), H = deposition distance of the granules (cm), t = time from the beginning of the collapse process (min).

The most common methods used in the sedimentation method can be listed as follows (Allen T., 1992).

- # hydrometer method
- # photostimulation method
- # sedimentation balance method
- # centrifugation method
- # x-ray absorption method
- # light diffraction method
- # current sensitive region method
- # methods based on flow dynamics
- # drag method

3. CONCLUSIONS

Our analysis technique differs according to the measurement range of the material. Furthermore, the definition of grain size in each analytical technique is determined by different methods.

- In sieve analysis, the measurement range is> 10 μm and the grain size is determined by the weight of the material passing through the sieve opening.
- In the microscope analysis; optic and electron microscopes respectively; the measurement range is 0.5-100 μm, 0.002-15 μm, and the grain size for both microscope types is determined according to Martin's, Feret's and equivalent circular diameter.
- Measurement range in laser diffraction analysis 0.05-2000 μm, specification of grain size according to equivalent spherical diameter,
- In electrical resistance analysis; the measurement range is 1-100 μm and the particle size is defined according to the equivalent spherical diameter,
- For sedimentation analysis is 1-75 μm and the grain size is defined according to the equivalent spherical diameter.

If particle size analysis methods are compared; laser diffraction analysis is fast and easy to use, electrical resistance analysis has a high resolution, and sedimentation analysis is better than others in terms of grain size.

According to this, the most reliable results are obtained in the electrical resistance method and the fastest analysis is obtained in the laser diffraction. Cost can vary greatly according to the manufacturer for the same technique. However, the most expensive method is again laser diffraction.

Since each method described above is based on a different physical property, it cannot be said that one has done a more accurate analysis than the others. It is also not possible to achieve such a result when the manufacturers are working on the development of these devices and the scientific researches carried out in these fields are taken into consideration.

Note:

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