

Particle Size Distribution Methods as adopted for different Materials

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Abstract: Information on particle size, shape, porosity and pore distribution of constituent materials are very important for choice and their appropriate use in construction. The known methods available for particle size distribution (PSD) are appropriate for materials of specific size range, shape, nature and distribution with improved techniques emerging with the advent of computer application in construction. Analysis of materials of varied grain sizes, shape, nature done by three methods of particular PSD (mechanical sieving, laser diffraction and CT-scanning) were reported in this paper for an assessment of their appropriateness and effectiveness for specific material. The study revealed mechanical sieving as effective for granular materials of 45 μm to 125 mm size range, the laser diffraction noted to be suitable for materials of fine particles but gives inadequate results when adopted for very fine materials susceptible to particle agglomeration. The CT- scanning on the other hand is noted to be adequate for PSD analysis of materials having medium to coarse size classification giving more definitive data on actual shape, distribution and volume of the material's particle. CT-scanning also gave results that are comparable to the laser PSD analysis when adopted for finer materials. The study concludes that required details and specific characteristics of the materials of concern should govern the choice of PSD methods and a call is made for good knowledge of available techniques by operators and investments on digital and recent equipment by our Institutions for improved outputs in materials research.

Keywords: particle size distribution, mechanical sieving, image analysis, CT-scanning and concrete constituent materials.

1.0 Introduction:

Particle size distribution (PSD) has been known to be of importance in the way the material performs and this is critical in use mix proportioning of concrete, while the new development of high-performance concrete (HPC) and ultra – high performance concrete (UHPC) has brought about the

need for having many constituent materials of varied sizes (some as supplementary cementitious materials and others as fillers) to be combined for an appropriate particle parking. The level of information required on these particles and their fineness level calls for adoption of more efficient approach to particle size analysis than what can be achieved from the conventional sieve analysis. Laser diffraction, scanning electron microscopy (SEM) and X-ray computed tomography scanning (CT-scanning) in the recent times are some of the approaches been adopted for materials characterisation. In these approaches, data for different particle sizes within a sample are captured at the same time by light, electron or x-ray emission and the data are processed to produce a PSD. This paper presents the report of three PSD analysis approaches (mechanical sieving, laser diffraction and CT-scanning) conducted on some constituent materials of concrete with a view to outlining the appropriateness of particular method for specific material as influenced by their nature, properties and fineness.

1.1 Sieve Analysis

Sieve analysis (also known as gradation test) is a simple technique and possibly the most common procedure in use for assessing the particle size distribution of granular materials. It is used for determination of the relative proportions of different grain sizes that make up a given soil/material mass. The analysis can be wet or dry while the sieving methods can be further classified as throw-action, horizontal, tapping, super-sonic, wet and air circular jet given the nomenclature as the specific activities required for effectiveness of the sieve operations. The test may be performed on many types of materials of non-organic or organic nature including sand, crushed rock, clays, granite, feldspars, coal, soil, a wide range of manufactured powders, grain and seeds, down to a minimum size depending on the exact method.

It entails allowing particles to pass through stack of sieves with known opening sizes and shaken for 10 minutes as recommended by Bowles (1992), using a mechanical test sieve shaker (Figure 1).



Figure 1: Set of Sieve on a horizontal sieve shaker AS 400 control (copyright Retsch Ltd.)

The sieves then removed from the shaker and weight of each sieve with the sample retained taken to the nearest 0.1 g. The mass of sample retained on each sieve is then obtained by subtracting the respective mass of each sieve (BS EN 933 Pt. 1: 1997) and the percentage passing calculated by subtracting the cumulative percent retained from one hundred percent (100%). From the calculated results, a semi-logarithmic curve is plotted with the ordinary axis (arithmetic) being the percent fines and the aperture size as abscissa (logarithmic scale).

Further quantitative analysis of the slope and shape of the PSD curve is done by means of the geometric values termed as the coefficient of uniformity (C_u) and the coefficient of curvature (C_c). The coefficient of uniformity (C_u) and the coefficient of curvature (C_c) can be expressed mathematically as:

$$C_u = \frac{D_{60}}{D_{10}} \text{ and } C_c = \frac{(D_{30})^2}{(D_{60} * D_{10})} \quad (1)$$

Where, D_{10} is the grain diameter (mm) corresponding to 10 percent passing on the PSD curve. D_{30} is the grain diameter (mm) corresponding to 30 percent passing on the curve. D_{60} is the grain diameter (mm) corresponding to 60 percent passing on the PSD curve (ASTM D421/422 2012; Vandavelde, 2008).

Atkinson (1993), Neville and Brooks (2002) and Shetty (2004) present “Fineness Modulus (FM)” as a ready index of coarseness or fineness of a material. It is the empirical factor obtained by adding the cumulative percentages of materials retained on each standard Sieve ranging from 80 mm to 150 μ m and dividing this sum by an arbitrary number 100.

The main limitation of mechanical sieving is the range of particle sizes that can be analysed often 45 μ m to 125 mm which is makes it ineffective for materials of very fine grains such as cement and other supplementary cementitious materials (SCM).

1.2 Laser Diffraction

In laser diffraction, particle size is calculated from the collection of light intensity data by a detector. The passage of the laser beam is through the sample particle at many different angles from the axis of the laser beam, as depicted in Figure 2. Fraunhofer diffraction and Mie theory of light scattering are the common diffraction theories used in particle size analysis by laser diffraction. Both theories claim that “the particle dimension is the optical spherical diameter” (Di Stefano et al. 2010).

Although laser diffraction is widely used, due to its simplicity and the capability of measuring a wide range of particle sizes (with the smallest particle limit as 1 μ m and the largest particle limit as 600 μ m). Kowalenko & Babuin (2013) and Di Stefano et al. (2010) argued that the technique has some inherent factors limiting its performance. Kowalenko & Babuin (2013) concluded from their research conducted on five different types of soils with a wide range of textures, that for a given

weight of sample, the sample's distribution becomes too large to employ the use of the modern laser diffraction instrumentation for particle size analysis as the change in particle geometry decreases. To support this argument, Burma et al. (1997) proposed that another limitation in the laser soil particle sizing is the accuracy of the optical parameters available for soil particles. This limitation leads to the under-estimation of clay particles sizes, although studies conducted using laser particle sizing produce repeatable results.

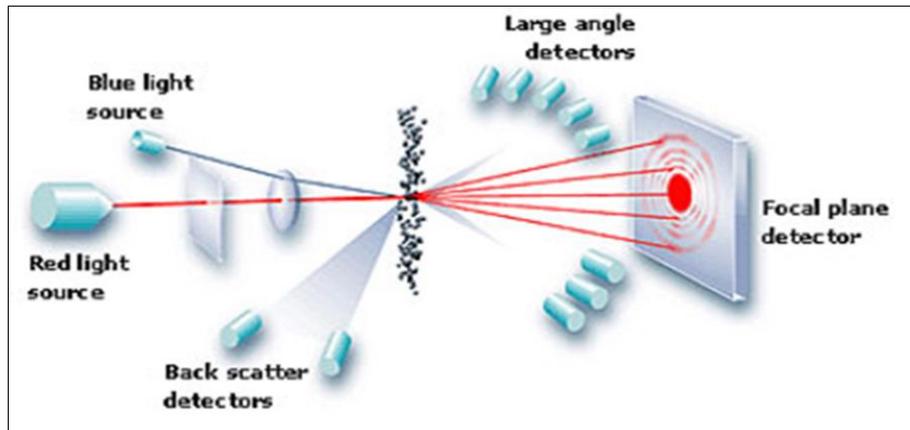


Figure 2: Principle of a laser diffraction (copyright Malvern Instruments Ltd.)

The identified issues with the conventional sieve analysis and laser diffraction for PSD has brought to fore the porosity and pore size distribution approach. Porosity is the measurement of voids between the particles or grains of a material. The porosity and pore size distribution are a function of the pore geometry which is directly affected by the packing density, particle size, particle shape, and cementation. Several studies have been conducted to quantify porosity and pore size distribution of different materials such as soil, cement, wood and metals using techniques including water retention curves, mercury intrusion porosimetry (MIP), tomography, nitrogen adsorption, and microscopy, but all these techniques have their limitations (Abell et al. 1999; Nimmo 2004; Romero & Simms 2009). However, to determine the geometric properties of pores, Abell et al. (1999); Nimmo (2004), and Romero & Simms (2009), suggested the application of image analysis using microscopes and tomography.

Abell et al. (1999); Nimmo (2004) and Romero & Simms (2009) categorically states that MIP should be coupled with an image analysis to establish a better understanding of porosity and pore distribution.

1.3 X-ray CT-scanning

X-ray CT-scanning involves the visualization of the internal structure of objects without sacrificing it. CT-scanning has been used in the medical field for several decades with technological advancement leading to its continuously growing usage as an analytical tool in the civil engineering industry (Asante, 2015). Although there is a difference in the conventional medical CT and industrial CT (e.g.

Micro CT); the technical principles are the same for the two. Data acquisition and image reconstruction are the two major processes. Image reconstruction refers to the conversion of the measured X-ray CT signals to a two-dimensional (2D) or three-dimensional (3D) image. Various mathematical procedures are employed (e.g., “Filtered back projection”, the “Feldkamp algorithm,” or Fourier-transform methods) depending on the technique and instrument used.

Specimen scanning by CT involves taking photographs of the sample from multiple angles by exposure to X-ray beams. An X-ray involves the penetration of various materials using the ability of electromagnetic radiation (high-energy photons). When an X-ray beam is directed to a material, part of their energy is either scattered, absorbed, or will travel through the material without any interaction with the material particles (Russ, 2002).

The thickness, density, and atomic number of the material, coupled with the energy of the photons, greatly affect the amount of X-ray transmitted, as shown in the Figure 2 below. For example, a dense object (e.g. metal and rock) absorbs more rays than less dense materials such as plastics.

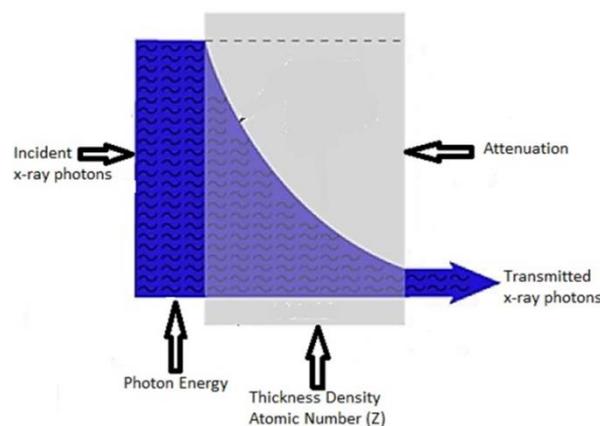


Figure 3: Factors affecting the transmission of X-ray through a material (Sprawls, 1995)

The purpose of radiography is to obtain a detailed image of the internal structure of an object. Radiography requires careful control of a number of different variables. A single image is not sufficient to give a description of an object’s internal structure.

Data collection generally occurs after the visualization of an object image file, which is visualized and analysed using a wide variety of 2D and 3D-based image rendering software.

The CT data collection depends on many variables, which includes; the number of views and the signal acquisition time per view. To capture a view, scanning can be done either by half rotation (180°) or full rotation (360°) at a closely or widely spaced view. A more closely spaced view yields finer image resolution and vice versa (Russ, 2002).

2.0 Materials and Methods:

Undisturbed soil samples were collected from burrow pits in Stellenbosch environment and made to undergo sieving and sedimentation (mechanical sieving) in accordance with ASTM D421/422 (2012) for PSD analysis. The clay fraction within the soil samples was determined by hydrometer analysis using 50g of the material passing through the number two hundred sieve (0.075 mm) and was dispersed for twenty-four hours in a one hundred and twenty-five millilitres (125 ml) solution of sodium hexametaphosphate and deionized water. The resulting solution was then thoroughly mixed and poured into a jar up to the one thousand millilitres (1000 ml) mark. The percentage passing was then calculated by subtracting the cumulative percent retained from one hundred percent (100%). The calculated results was plotted on a semi-logarithmic curve with the ordinary axis (arithmetic) being the percent fines and the aperture size as abscissa (logarithmic scale) as shown in Figure 4 (mechanical sieving). Some portion of the soil sample was also analysed with the CT-scanning and the result plotted on the same semi-logarithmic curve (Figure 4 – CT-scanning) for comparative study of the output from the two methods.

Laser diffraction analysis was carried out on six different fine particles comprising binders - CEM I 52.5 N supplied by Pretoria Portland Cement (PPC), Western Cape, South Africa conforming to BS EN 197-1-2000 and SANS 50197-1; silica fume (SF) by SiliconSmelters of the FerroAltantica group; fly ash (FA) from AshResources; corex slag (CS) supplied by PPC and two size classifications of superabsorbent polymers (SAP) – a concrete internal curing agent (i.e. FLOSET CS 27 (< 300 μm) and FLOSET CC 27 (< 600 μm) supplied by SNF Floerger, France. The two size classifications of SAP was also analysed for PSD with the CT-scanning for a comparative study of the Laser diffraction and the CT-scanning methods.

Analysis of the binders and SAP particles through the Laser diffraction for PSD adopted the use of a Saturn DigiSizer 5200 V1.10 (5200 LSHU V2.01 S/N 216) high definition digital particle size analyser with a Mie model, while specific surface area in nitrogen (N_2) adsorptive medium was also determined using 3Flex (Version 1.02 and S/N 103) surface characterisation (both equipment made by Micrometrics Instruments Corporation). The Laser diffraction particle size analysis was by a wet technique using Isopropanol (an ethanol non-absorbent liquid medium) at a Refractive Index (RI) of 1.376 with a Laser 658 nm light source giving analysed particle range specification of 0.1 to 1000 μm up to a lower limit of 0.07 μm on the instrument. The instrument has a 600 ml reservoir at a 1.2 litre/min pump speed.

Analysing the grain sizes and distribution of the soil and SAP particles by CT-scanning involve scanning to obtain 3D X-ray images using a General Electric Phoenix VTomeX L240 X-ray microCT scanner. The 3D X-ray images were then examined and analysed using Avizo Fire, version 8.0 by FEI

Visualisation Science Group and VG Studio Max 2.2 by Volume Graphics to filter and classify the individual grains for determination of the sizes and their distribution.

3.0 Results and Discussion:

The plot of the PSD of the soil samples by the mechanical sieving and CT-scanning method is shown in Figure 4 while Table 1 gives a summary of the characteristics. The PSD result from the CT-scanning differs slightly from that obtained from mechanical sieving.

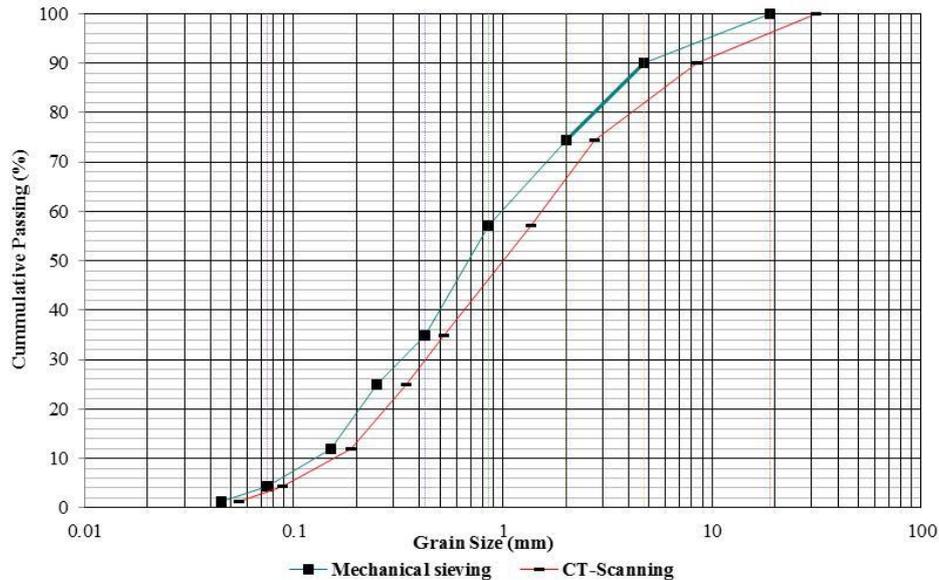


Figure 4: Comparative plot of PSD by Mechanical Sieving and CT-Scanning (Asante, 2015)

Table 1: Summary of PSD for Soil Sample by Mechanical Sieving and CT-Scanning (Asante, 2016)

Characteristics	diameter (mm)	
	MS*	CT*
D ₉₀	4.75	8.50
D ₆₀	0.85	1.70
D ₅₀	0.78	1.00
D ₃₀	0.33	0.40
D ₁₀	0.14	0.18
C _u	6.07	9.44
C _c	0.92	0.52

*MS = Mechanical Sieving; *CT = CT-scanning

The CT-scan measured particle size in Figure 4 seems larger than the measured mechanical sieve particle size. This occurs when very fine particles (silt) clump together and are then sent in a digital image as a single homogenous particle. This makes individual particles blurry. The parameters retrieved from CT-scan images using Avizo Fire version 8.0 includes the volume and area of the individual particles within the region of interest (ROI). The actual diameter of each particle/grain can be calculated from either the volume or area as obtained from Avizo Fire version 8.0. Therefore, CT-scan measured coarse particles are more accurate than particles measured in mechanical sieving,

which operates on a mass-based result instead of particle diameter. On the other end, the measured particle size for finer particles is more accurate in mechanical sieving than in CT-scanning. PSD of finer material (<150 μm) when done by dry sieving will yield significantly less accurate results due to the fact that, in sieve analysis, particles are assumed spherical and this is not true for all particles. Needle or rod like particles will either pass through the sieve or remain behind on the sieve, depending on its orientation.

PSD for medium to coarse particles is observed as more accurate in CT-scan than in mechanical sieving while, the measured PSD for finer particles on the other hand can be more accurate in mechanical sieving than CT-scanning depending of the minimum pixel size of scanning in the CT.

Figure 5 (PSD plot) and Table 2 gives a summary of the PSD and specific surface area of cementitious materials (CEM I 52.5 N, SF, FA and CS) and dry SAP particles (SP₁ and SP₂) carried out by laser diffraction PSD method and Brunauer–Emmett–Teller (BET) nitrogen absorption technique analysis.

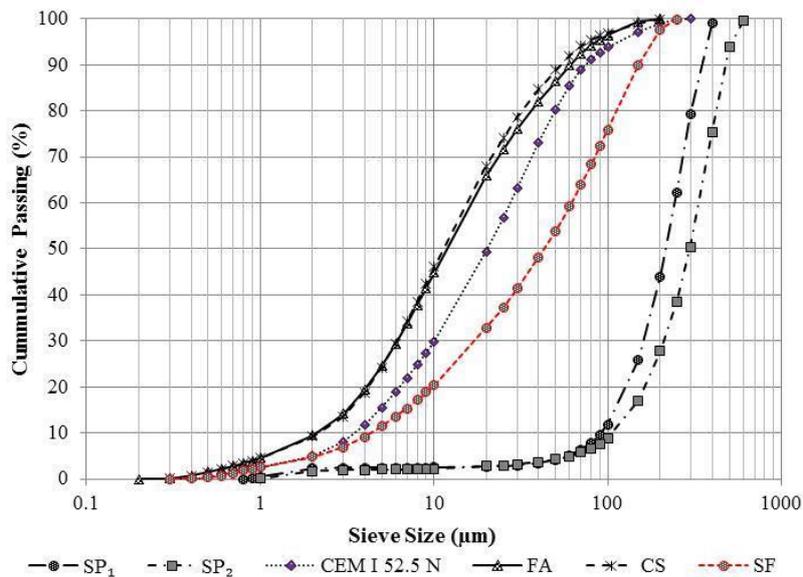


Figure 5: PSD analysis of various fine materials by Laser Diffraction (Olawuyi, 2015)

Table 2: Summary of Laser Diffraction PSD of Binders and SAP (Olawuyi, 2015)

Characteristics	diameter (μm)					
	SP ₁	SP ₂	CEM I 52.5N	SF	FA	CS
D ₉₀	337.8	472.4	73.7	150.3	60.3	53.2
D ₅₀	216.1	298.4	20.4	43.2	11.7	11.2
D ₁₀	91.8	108.1	3.5	4.4	2.1	2.2
Mean	214.9	293.8	32.9	61.5	23.0	21.6
Median	216.1	298.4	20.4	43.2	11.7	11.2
Model	300.4	400.5	30.0	112.9	11.3	11.3
BET surface area (m^2/g)	3.82	4.45	5.56	19.16	5.13	3.22
Model	(1.43, 0.001), 1.376	(1.43, 0.001), 1.376	(1.72, 0.100), 1.376	(1.52, 0.100), 1.359	(1.45, 1.000), 1.376	(1.45, 1.000), 1.376

The laser diffraction PSD was repeated and similar result obtained with the grain size of SF presented (red colour on Figure 5 and Table 2) as larger than other cementitious materials (CM).

The results show that the binders are fine in nature with the CEM I 52.5 N, FA and CS having over 90% (D_{90}) of the particles below 75 μm size and 50% (D_{50}) of the particles being lower than 25 μm (CEM I 52.5 N) and 12 μm in size (FA and CS) respectively.

SF is reported from the laser diffraction PSD as the coarsest of all the cementitious materials (CM). It has D_{50} of 43 μm and D_{90} of 150 μm but BET specific surface area is 19 m^2/g . This is about five times the BET specific surface area of other cementitious materials (CEMI – 5.56 m^2/g , FA – 5.13 m^2/g and CS – 3.22 m^2/g). This affirms that SF is actually much finer than the other the three CM. The BET method is recommended in literature (Silica Fume Association, 2005) as the best approach for determining the specific surface area of SF while laser or other PSD methods may be adopted for other CM; specific surface area for SF particles is often determined by the BET method. Neville (2012) is of the view that absolute measurement of specific surface area can be obtained by BET – nitrogen adsorption method as the internal area of the particles is also accessible to the nitrogen molecules.

The Laser diffraction PSD result for grain sizes of SF reported in Table 2 and Figure 5 (red colour) can be adjudged as incorrect. The grains sizes in SF are expected to be much lower in value (about 100 to 150 times finer than CEM I 52.5 N), typically < 1 μm . Reports from literature (Siddique & Khan, 2011) gave an average particle size of 150 nm. It should be of note that the SF came out of the bag in the condensed form and effort made at grinding with mortar and pestle in the laboratory before the laser diffraction PSD test, possibly did not result in complete separation of the grains. This could explain the error in the result of the laser diffraction PSD test on SF. The Laser diffraction method is reported by (DiStefano, et al. 2010; Kowalenko & Babuin, 2013) as having some inherent errors in determining particle sizes of very fine materials susceptible to particle aggregation.

The SAP particles were observed to be coarser than the CM with SAP type II (SP_2) having grains ranging between 0 and 600 μm in size, D_{90} and D_{50} of 472.4 μm and 298.4 μm respectively, being the coarsest. SAP type I (SP_1) has particle sizes between 0 and 400 μm sizes, D_{90} and D_{50} of 338 μm and 216 μm . BET specific surface area values - 3.82 m^2/g (SP_1) and 4.52 m^2/g (SP_2) respectively are similar but a bit lower than that of CEM I 52.5 N, FA and CS.

The CT-scanning analysis of the SAP particles sizes and distribution (Figure 6 and Table 3) gave similar result as the Laser diffraction. SP_1 has particle sizes within 3 and 300 μm (i.e. < 300 μm), while SP_2 has particles ranging between 3 and 600 μm (i.e. < 600 μm) as presented in Figure 6.



Figure 6: PSD analysis of SP₁ and SP₂ by CT-scanning (Olawuyi, 2015)

The D₉₀ and D₅₀ for SP₂ as shown in Table 3 are 400 μm and 270 μm against the earlier Laser diffraction values of 472.4 μm and 298.4 μm. SP₁ has D₉₀ (= 250 μm) and D₅₀ (= 170 μm) as against the 338 μm and 216 μm gotten from Laser diffraction. The little differences can be accounted for by the image reconstruction and data processing approach employed. The issue for further study is the level of accuracy to which CT-scanning can be for analysing materials of very fine nature like the SF.

Table 3: Summary of PSD of SAP determined by CT-scanning (Olawuyi, 2015)

Characteristics	diameter (μm)	
	SP ₁	SP ₂
D ₉₀	250	400
D ₆₀	180	300
D ₅₀	170	270
D ₃₀	140	195
D ₁₀	90	130
C _u	2.00	2.31
C _c	1.21	0.98

4.0 Conclusion and Recommendations

This study revealed that the choice of particular methods of particle size distribution should be governed by the details required and specific characteristics of the material to be analysed. Mechanical sieving though simple and easy, is only suitable for granular materials while little error may arise in size classification and distribution caused by shape and nature of the material sample. The use of laser diffraction and image analysis may serve as better alternatives and improved method of PSD with resultant more detailed characteristic data such as pore size, porosity and pore distribution possible. Particle agglomeration and disintegration of some materials under light or image data acquisition and processes calls for some caution. Good knowledge of the available techniques and investing in digital and recent equipment will enhance more reliable output in materials research

and usage in Building and Construction works. The following inferences and recommendations are thereby offered:

- i. Particle size distribution by the mechanical sieving method should be restricted to granular materials and soil samples of grain sizes above 150 μm .
- ii. Laser diffraction when adopted for fine materials such as cement and other cementitious materials should give consideration to compatibility of the material to the dispersing medium while the proper refractive index for the particular material should be known. Analysing very fine materials requires determination of the specific surface area of the materials possibly through the BET approach.
- iii. PSD analysis by CT-scanning (at both Micro and Nano-image analysis levels) should be given more attention in future studies with a view at establishing possible alternative method suitable for wider particle size range.
- iv. Universities and Research Institute in Nigeria should invest more in the specialist training and development of the technical staff and acquisition of new improved facilities and equipment for better quality of research targeted at addressing specific needs of our environment.

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