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Synthetic Determination of Specific Density, Specific Surface Area and Particle Size Distribution of Cementitious Powder Materials

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Abstract

The use of cementitious materials for construction of buildings and structures started during ancient civilization. Science of modifying physical and chemical properties of cementitious materials is a continual process which is because of a need to increase reactivity and improve strength and durability properties of binders. Due to increasing demand of modern infrastructures and continual depleting of binder sources the scientist, engineers and researchers work hard on improving binding properties of cementitious materials for construction of low cost and durable structures. Among of the factors which affect binding properties of cementitious properties are densities, surface areas and particle size distribution. Several methods and procedures have been developed to determine these physical properties on which other require huge capital investment and others takes long time to complete a test which hinders further investigation and improvement of alternative binders. This study investigated that there exist an 'S' curve similar to particle size distribution curve when time air flow against weights of sample measured using Blaine apparatus is drawn.

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The gradients of lower and middle lines of the curve and values at turning points were synthesized to determine specific densities, specific surface areas and particle size distribution of powder materials. The approach is time saving and the equipment is affordable by many researchers. In order to have accurate results the approach can be automated.

Keywords: Synthetic; cementitious; densities; surface areas; particle size distribution; packing density; reactivity.

1. Introduction

Cementitious materials are used to binder other materials such as aggregates, sands, bricks, blocks and stones for construction of durable and cost effective buildings and structures. Cementitious materials are ancient binders for construction works, Romans, Greeks, Egyptians used pozzolanic powder materials as binders for construction of houses and other structures 300AD [1]. The binding strength of cementitious powder materials depends on content of active minerals and physical properties of binders [2]. The basic physical properties of powder binders which have great influence to strength and durability properties of the cured specimens are specific surface area, densities and particle size distribution.

a. Effects of density on properties of cementitious binders

Density is the physical property which depends on chemical composition, molecular arrangement and morphological characteristics of materials which govern the weight of a fixed volume of the sample materials. There is wide range of densities for cementitious materials which is due to variation of porosities, grain shapes, mineral contents and composition. Addition of admixtures to cementitious materials such as OPC or blending together cementitious materials improves strength and durability resistance of specimens. Blending of material ingredients can be by mass or volume batching. The contents of admixture may be higher than that of replaced cement if density of admixture is lower than cement which may affect the reaction behavior and mechanical properties of the hardened specimens [3]. Studies have recommended proportioning of material ingredient by volume when materials having higher differences in densities are blended for making binders [4, 5]. This is because weight batching causes more volume for low density materials which affects properties of fresh mixtures and hardened specimens such as flow, water binder ratio, densities of cured specimens, reaction characteristics and finally strength development and durability resistance of cured specimens. However specific density of binder affects specific surface area of materials. Specific density is the parameter for determination of specific surface area [6]. Materials with high specific density will result into low specific surface area regardless of having the same mean particle sizes and particle size distribution. Specific surface area and particle grain size directly affects reaction of binders, high specific surface area high reactivity and smaller grain size high reactivity of materials [2].

b. Effects of specific surface areas on properties of cementitious binders

Fineness of materials accelerates chemical interaction of materials which affects hydration and/or pozzolanic reaction of binders [2]. High specific surface area improves compressive strength of cured specimens, durability

and rheological characteristics of fresh mixtures [7, 8, 9, 10]. This is because finer particles exposes nucleus of the grains, which enable fast dissolution of reactive elements to take place and enhanced hydration and pozzolanic reaction of the mixtures [2]. By increasing specific surface areas of materials results into increased cost of production of binders since more energy and time is required to grind the materials [11]. But also high fineness increases water cement ratio for consistency and workability of binders because more water is required to lubricate the surfaces of particle grains [2]. However finer particle size of powder binders enhance packing densities of the mixtures which increases densification hence increased strength and durability of the concrete and mortars [7]. The specific surface areas of binders are widely determined using Blaine method. The method involves determination of weight of sample required to form a well compacted bed from specific density as given in equation 1. A time air flow through a compacted sample bed in Blaine tin is determined which is used to compute specific surface area of material as given in equation 2 [6].

$$M_s = \frac{\rho V}{2} \quad 1$$

Where: M_s – weight of sample to fill half of a tin volume (gm)

V – volume of a tin (cm^3)

ρ – density of a sample (gm/cm^3)

$$SSA = \frac{K}{\rho} \times \frac{\sqrt{e^3}}{(1-e)} \times \frac{\sqrt{t}}{\sqrt{\eta}} \quad 2$$

Where: SSA – specific surface area (cm^2/gm)

K – apparatus constant at room temperature and humidity

e – porosity of the cement bed (0.500)

t – time of air flow measured between etched lines (sec)

η – viscosity of air at test temperature and humidity (Pa.s)

Several factors can increase specific surface areas measured using Blaine approach regardless of having the same PSD and mean particle size. Among of the factors are densities of materials, temperature variation, volume of materials, pressing effort and time and variation of moisture content [2]. The increased moisture content of materials increases specific surface area of the sample, this is because moist materials enhance compaction process and limit air flow through sample bed.

In order to have accurate results a constant pressing effort of the plunger for all measurements should be applied but also the sample materials should be dried to constant moisture content. To minimize errors due to measurements of weights and variation of pressing efforts the process can be automated. This is because a very small change of weight of sample within the Blaine tin has big impact on computed specific surface areas [2].

c. Effects of particle size distribution on properties of cementitious binders

The particle size distribution of binder materials significantly affects properties of fresh and hardened mixtures. Uniformly graded particle sizes improve flow characteristics of fresh pastes, mortars and concrete, improves packing density, and compressive strength development of the mixtures [7].

Investigation made by Chengula [2] on supplementary cementitious materials found that the PSD of binders should lay within a specified envelopes. High compressive strength of cured paste specimens were achieved when the mean particle sizes of powder materials were within a determined range. In order the binders to achieve high reactivity, maximum stiffness index and maximum compressive strength of the mixtures he suggested that the distribution of binder powder material should lay within the proposed envelopes. However there is no direct relationship between particle size distributions and specific surface areas. The binder powder material can have the same specific surface area but with different particle size distributions which can influence physical and chemical characteristics of fresh and hardened mixtures.

Particle size distribution affects interlocking behavior and friction resistance of the matrix which greatly influence improvement of compressive strength and durability resistance of the cured specimens [2].

Chengula [2] suggested that the best measure of fineness of powder materials as binders is the combination of particle size distribution (PSD) with mean particle sizes. This is because the chemical reaction of binders is greatly influenced by the size and distribution of grains. Grain sizes together with PSD are affected less by other factors than specific surface area [2].

2. Investigation procedure

Investigation for this study involved milling of different cementitious materials and determining specific densities and surface areas.

Four sample materials, pozzolan, limestone, gypsum and CEM I 42.5N were used for investigation. The samples were milled using ball mill at various milling duration to investigate effect of fineness of materials on densities and specific surface areas of samples. The densities of the samples were measured by using Helium Pycnometer (Accupyc 1330, Fa. Micromeritics) and specific surface areas of the samples were determined using Blaine air permeability apparatus following the procedure stipulated in BS EN 196-6 [6].

a. Investigation approach

The milled samples were filled in a prepared sample bed of Blaine tin. The samples were measured in weight using electronic balance at constant intervals.

Cumulative weights of sample were measured for time air flow using Blaine air permeability apparatus. The graphs of time of air flow versus weights of the sample were drawn. Figure 1 is the typical curve of time air flow versus weights of sample for powder materials.

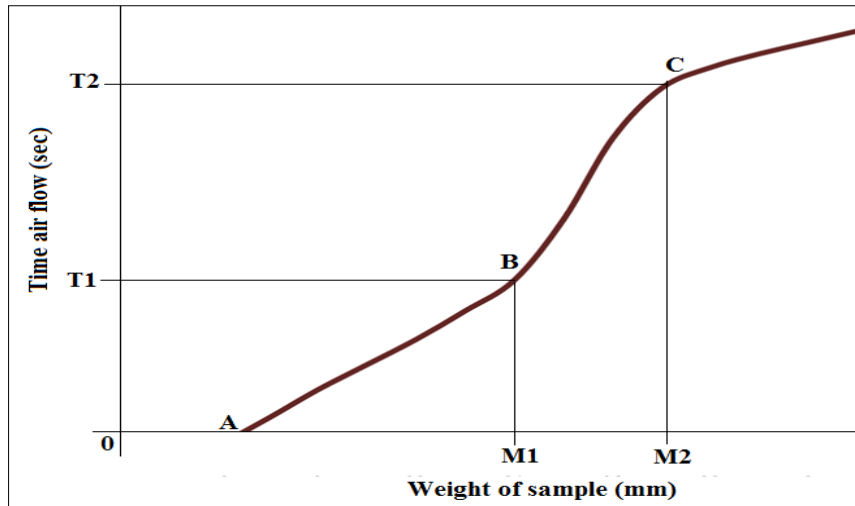


Figure 1: Graph representing time air flow versus weight of sample in a tin

The graph indicates an ‘S’ curve similar to cumulative particle size distribution curve. The curve consists of three lines which are lower part of the curve which starts at zero time air flow the point intersecting the horizontal axis to first turning point T_1M_1 , the middle part which lay between two turning points T_1M_1 and T_2M_2 and upper part which starts at point T_2M_2 . The middle part have steeper slope compared to lower and upper part of the curve. Investigation made by this study revealed that the weights for determination of specific densities and surface areas lay within middle part of the curve.

3. Results and discussion

This study presents synthetic determination of specific densities, surface areas, and particle size distribution of powder materials. These properties of cementitious materials have great importance to hydration and pozzolanic reaction of binders which affects mechanical and durability properties of mortars and concretes for construction of buildings and structures [2]. It is important to determine these physical binder properties as they affect blending design of ingredients and reaction mechanism of the cementitious materials [2].

a. Synthetic specific density of powder materials

The specific density of powder binder material is computed by dividing weight of sample to void free volume occupied by the sample. The specific densities of powdered materials can be determined using Helium pycnometer density equipment and liquid glass bottle pycnometer apparatus. Investigation made by Munro and his colleagues [12] indicated variation of densities measured using the same approach and using different approaches for the same sample materials. Several factors can cause variations of densities which include variation in temperature and pressure, moisture contents, contaminants and porosities.

The investigation made by this study found that the weights of the sample computed from specific densities determined by using Helium pycnometer equipment were found to lay within the two turning points of the curve (figure 1). The weight of sample to produce optimum compacted sample bed for measuring specific surface area

of powder material is given by equation 1 [6].

By using the weights of the sample at turning points and standard volume of Blaine tin, the specific densities of powder materials can be determined from equation 3.

$$\rho = \frac{2M_s}{V} \quad 3$$

Where: ρ – specific density of powder material (gm/cm³)

M_s – weight of sample at turning points (gm)

V – volume of Blaine tin (1.628cm³)

Care should be taken during measurements and pressing efforts of the plunger. In order to get precise results, the weights of the sample should be measured at constant intervals but also pressing effort of plunger should be constant for all measurements. Figure 2 shows ‘S’ curves of time air flow versus weights of samples for pozzolan, limestone, gypsum and CEM I 42.5N powder materials.

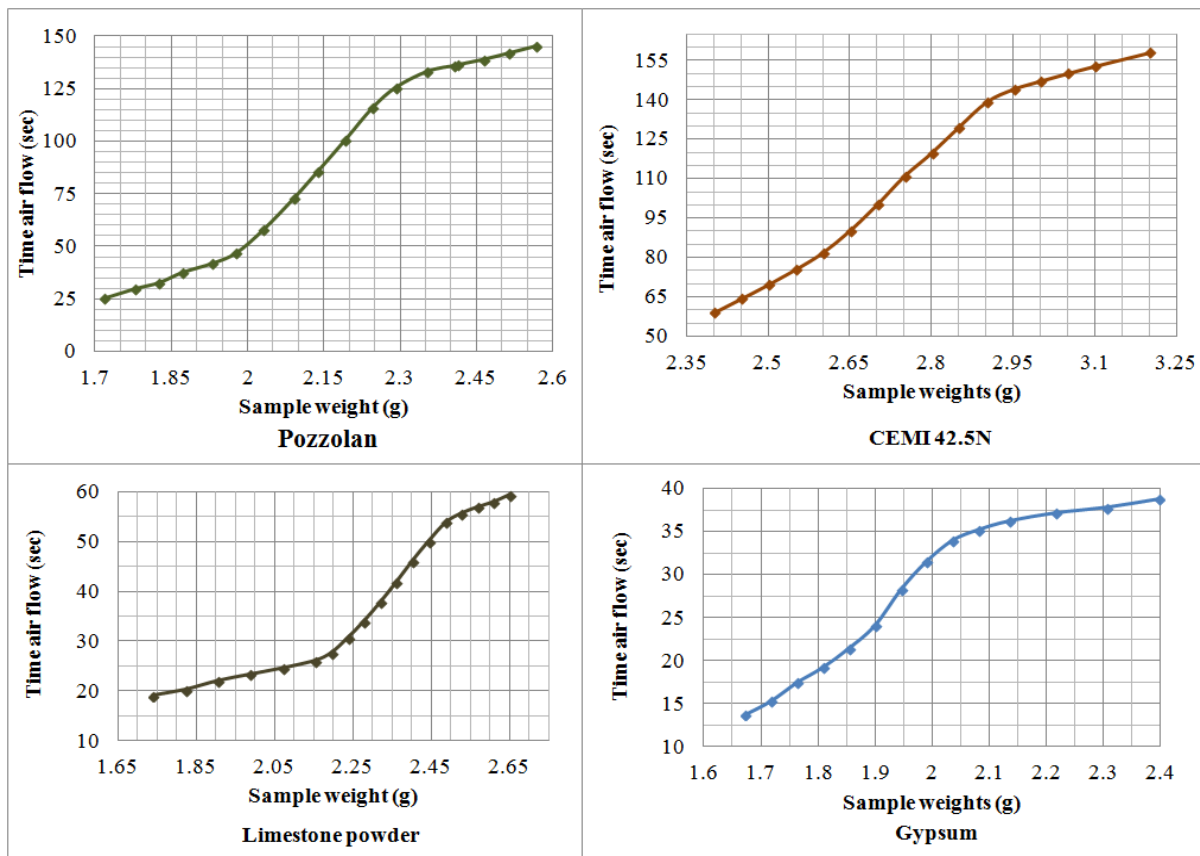


Figure 2: Graphs of time air flows versus weights of samples for various powder materials

The parameters used for determination of specific densities, specific surface areas and particle size distributions are obtained from the curves of time air flow versus weight of sample. Table 1 gives weights of samples at

turning points, time of air flow at turning points and gradient for lower and middle parts of the curves.

Table 1: Parameters of sample materials from graphs

Parameters	Sample materials			
	Pozzolan	Limestone	Gypsum	CEM 42.5N
M ₁ (g)	1.983	2.174	1.898	2.610
M ₂ (g)	2.294	2.487	2.058	2.902
T ₁ (sec)	48.010	26.860	24.060	81.779
T ₂ (sec)	125.790	53.830	34.550	139.230
m ₁ (sec/g)	82.760	17.040	42.500	113.510
m ₂ (sec/g)	270.810	94.720	82.360	194.340

b. Synthetic specific surface area of the powdered materials using Blaine apparatus

The specific surface areas of powdered materials are determined by using Blaine fineness approach, BET method and empirical equation built in laser diffraction particle analysers’ software (LD-PSD) and X-ray computed tomography (X-CT). Currently only BET is considered to provide the most fundamental surface area measurement since the method does not assume the particles are spherical [13]. The disadvantages of BET method is performed by using expensive device and takes long time to measure one sample but also gives much higher specific surface areas compared to other approaches [13].

However Blaine air permeability approach is widely used for determination of specific surface areas and is because of its simplicity and time serving [14, 15]. But also the test is conducted by using less expensive equipment which is affordable by researchers and scientists even in developing countries.

The specific surface areas of powdered materials determined by using Blaine approaches considers square root of time air flow as the major parameter of the equation [6, 16]. By considering time air flow as major parameter of the equation results into tremendous difference of specific surface area of the same sample at different measurements. This is because a very small change of weight of sample within the Blaine tin has big impact on computed specific surface areas. Little increase in weight of materials in Blaine tin increases specific surface areas significantly [2]. This is because weights of sample materials filled in Blaine tin for determination of specific surface areas lay within middle part of the curves which have steep slopes. Therefore small change in weight results into tremendous increase in specific surface area of the sample materials. The study on different samples conducted (figure 2) indicates an ‘S’ curve phenomena for time air flow versus weight of sample similar to particle size distribution curve.

However the specific surface area increases with increasing fineness of materials [2, 10, 13]. The fineness is increased by reducing grain particle sizes through grinding process of the sample materials. For this study the samples of pozzolan material were milled using ball mill machine for 10 minutes and 20 minutes respectively. The curves for time air flows versus weights of the samples were drawn (figure 3). The graph show that sample milled for 20 minutes have steeper slopes compared to the sample milled for 10 minutes. However the two

curves turns at the same weights regardless changes in slopes of lines.

This situation indicates that the weights at turning points of the curves are the properties of particular materials and are the points giving specific densities of materials.

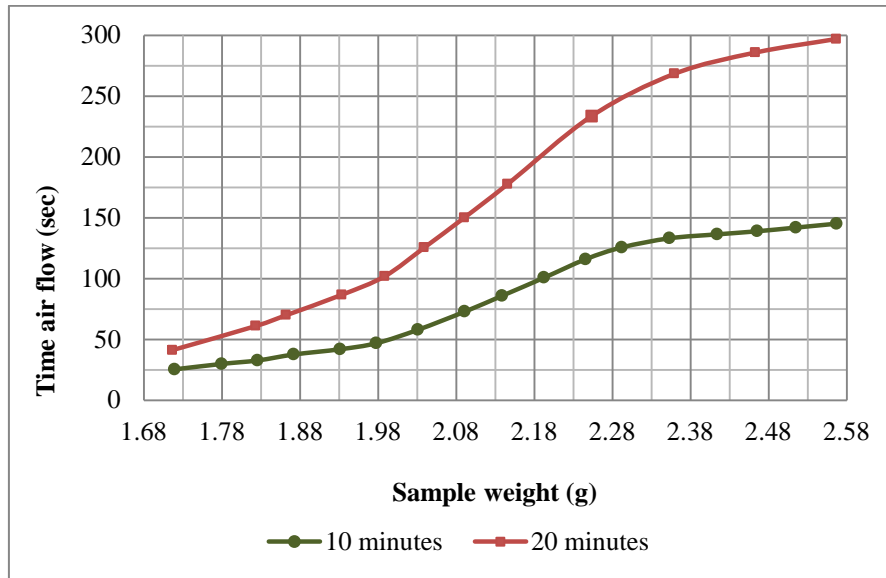


Figure 3: Graphs of time air flows versus weights of pozzolan at different milling duration

The change of slopes of the curves is due to the change of fineness of particle sizes of the materials keeping other factors constant. The increase in fineness of powder materials results into increasing slopes of the curves (figure 3). This indicates that specific surface area is proportional to the slopes of the lines of curve.

Equation 4 is the modification of equation 2 used to determine specific surface areas of powder materials using Blaine approach. Equation 4 has incorporated gradients (slopes) of lower and middle parts of the curve and this is because variations of gradient at range of data set are minimal. Therefore by using gradients of curves will affect less specific surface areas of sample materials than using time air flow.

$$SSA = K \cdot \frac{\sqrt{e^3} \cdot \sqrt{M}}{\rho_s \cdot (1-e)} \cdot \sqrt{\frac{m}{\eta_a}} \quad 4$$

Where: SSA – specific surface area (cm²/g)

m – curve gradient (slope) of time air flow versus weight of sample (s/g)

η_a – dynamic viscosity of air at test temperature and humidity

ρ_s – specific density of sample

e – porosity of sample bed (e = 0.5)

M – weight of sample at turning point(s) of curve

K – Equipment and measurement constant

The constant of the equation is to be determined from material with known specific surface area. The specific densities and surface areas of powder materials using equations 3 and 4 should be taken as average of the lower and middle part of the curve. Table 2 gives specific densities and specific surface areas of pozzolan, limestone, gypsum and CEM I 42.5N.

Table 2: Specific densities and surface areas of sample materials

Properties	Sample materials			
	Pozzolan	Limestone	Gypsum	CEM 42.5N
ρ_1 (g/cm ³)	2.436	2.671	2.332	3.206
ρ_2 (g/cm ³)	2.818	3.055	2.528	3.566
ρ_{ave} (g/cm ³)	2.627	2.863	2.430	3.386
SSA ₁ (cm ² /g)	3909.6	1694.1	2863.2	3991.3
SSA ₂ (cm ² /g)	6575.4	3734.9	3828.6	4950.9
SSA _{ave} (cm ² /g)	5242.5	2714.5	3345.9	4471.1

In order to have accurate results a constant pressing effort of the plunger for all measurements should be applied but also the sample materials should be dried to constant moisture content. To minimize errors due to measurements of weight and variation of pressing effort the process can be automated.

c. Synthetic particle size distribution

The particle-size distribution (PSD) of powder material is a list of values in percentages computed from fractions of masses or volumes or areas of individual particle size group of the total mass or volume or area of the sample [17, 18]. The particle size distribution analysis are used to express physical characteristics of materials such as particle median size, mean size, maximum and minimum sizes and distribution pattern. The particle size characteristics have great influence to particle packing behavior, chemical interaction of mixed materials, flow, mixing and compaction process, which then affect strength and durability of the mixtures (pastes, mortars and concretes) [2, 17, 19, 20].

Several techniques are used to determine particle size distribution such as sieving method, microscopy technique, liquid/gas sedimentation techniques, laser light scattering techniques, optical and electrical sensing zone method etc [14, 15, 21, 22, 23].

The choice of the method depends on several factors such as accuracy and precision of the results, solubility, reactivity and toxicity of the materials, duration to conduct the analysis and simplicity of the method, life cycle of equipment etc [24].

For this study particle size distribution curves were derived from relationship between gradients of the curves and weights of the sample measured using Blaine air permeability apparatus (figure 2).

Researchers indicated that fine particle sizes increase specific surface areas [10, 13] which are due to increased gradients of the curves.

Therefore particle grain sizes are inversely proportional to gradients of the curves keeping other factors constant.

The particle grain sizes were determined by considering weights of the sample and gradients of the lower and middle parts of the curve as indicated in equation 5.

The cumulative percentage passing is determined using equation 6.

$$G_{ps} = K \left[\frac{1}{\eta_a(m_1+m_2)} \right] * \left[\frac{M_i - \alpha M_o}{M_f - M_o} \right] \quad 5$$

Where: G_{ps} – grain particle sizes (μm)

K – constant of the equation, for this study a value of 13,650 was used

η_a – dynamic viscosity of air at test temperature

m_1 and m_2 – gradient of lower and middle curves (s/g)

M_o – weight of sample at zero time air flow (g)

M_i – weight of the sample for each measurement from M_o to M_f (g)

M_f – The weight of sample at second turning (g)

α – factor for sample weight at zero air flow (0.99)

The weights of the sample especially for lower part of the curve can be extrapolated to get other measurements to intersect horizontal axis at zero time air flow.

$$\%CP = 100 \frac{T_i}{T_f} \quad 6$$

Where: CP – cumulative passing (%)

T_i – time air flow at each measurement starting at zero time air flow to T_f (sec)

T_f – time air flow at second turning point (sec)

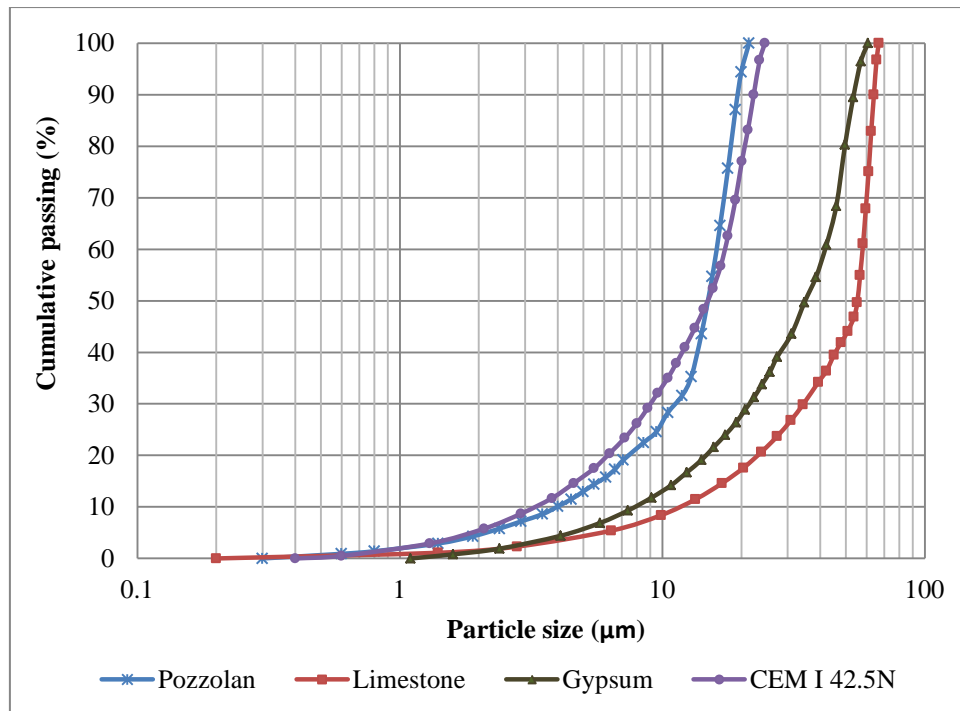


Figure 4: Synthetic particle size distribution of powder materials

Figure 4 shows synthetic particle size distribution curves of pozzolan, limestone, gypsum and CEM I 42.5N powder materials. The particle size distribution curves are useful information for comparison of particle size gradation, particle size parameters ie mean and median sizes but also the expected fineness of powder materials.

4. Conclusion and recommendation

The approach of using Blaine air permeability apparatus to determine specific densities, specific surface areas and particle size distribution of powder materials is simple to use, takes few time and the equipment is potable and affordable. The approach can be computerized to assist researchers and scientists especially in developing countries where it is difficult to afford cost of buying expensive equipments to determine specific densities, surface areas and draw particle size distribution of powder binders. It is necessary to determine these properties for all materials ingredient used for blending design of construction binders since on one way or another affects pozzolanic and hydration reactions.

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