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〈연구논문〉

Plate/Plate, Truncated Cone/Plate 센서를 이용한 시멘트반죽 유변학적 특성에 대한 개선된 측정

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Improved Measurements of the Rheological Property of Cement Paste Using Plate/Plate and Truncated Cone/Plate Sensors

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요 약

Plate/plate 및 Truncated cone/plate 감지부를 이용한 시멘트반죽의 유변학적 특성을 측정함에 있어, 관련된 오차에 대한 실험 및 이론적 분석이 보고된다. 주요인자로는 감지부 직경과 상하 감지부 사이의 간격이 고려되었다. Cone/plate 감지부의 원추끝을 깍아 간격의 크기를 늘린 Truncated cone/plate 감지부의 토르크 분포에 대한 이론적 분석결과 원추 간격이 0.7 mm까지 절단되더라도 8.5%의 오차범위내에서는 cone/plate 감지부의 역할을 수행하는 것으로 판단된다. 실험결과는 샘플주변의 상대습도가 결과에 영향을 끼치고, 감지부의 기하학적 요인에도 매우 민감함을 보여준다. 이 결과는 정지시간을 포함한 오랜 실험기간동안 미소변형하에서 유변학적 특성을 연구하는데 도움이 될 것으로 판단된다.

Abstract－The results from experimental and theoretical analyses of possible artifacts associated with measuring the rheological property of cement paste using plate/plate and truncated cone/plate sensors are reported. Important considerations include the sensor diameter and the size of a gap between sensors. A theoretical investigation shows that for a truncated cone/plate sensor, the error introduced by a truncated cone tip of 0.7 mm on torque is less than 8.5%. Experimental results show that the relative humidity around a specimen influences the results. The measured rheology of cement paste is sensitive to the geometry of a sensor, and to the ambient relative humidity. This study has a lead in small strain rate rheology under long experimental time and quiescence.

Keywords: Rheology, cement paste, truncated cone/plate, geometry of a sensor

1. Introduction

The rheological properties of cement paste have been studied extensively such as the time dependent behavior under constant shear rate and the dynamic behavior under varying shear rate, in which the frequency or the strain of oscillatory sensor was varied [1]. These experiments typically employed large deformations. The structure of cement paste, however, is broken under relatively small strain, and this destroys information about the undamaged structure, which must be investigated using small strain. Microstructure of cement paste within two hours after mixing of cement particle and water, keeps the inter-connection forces generated by thin films between water-coated cement particles. These forces can be quantified by measuring a yield stress at small strain region without breaking the undamaged structure. So far, yield stresses were obtained through a flow curve experiment performed by decreasing shear rates from large values to small values. Yield stresses at the flow experiment represent the microstructure of cement paste after the breakdown of inter-connection forces because they are defined at the stress axis by the extrapolation of a constant gradient line decided at large strain region. They were ten times lower than those measured by small strain experiment which could display the build-up structure of cement paste after mixing. Rheological behavior of cement paste has been studied through a plot of shear stress vs. shear strain under very small shear rates as shown in Fig. 1 [2]. Shear strain was obtained by integration of shear rate. At small deformation

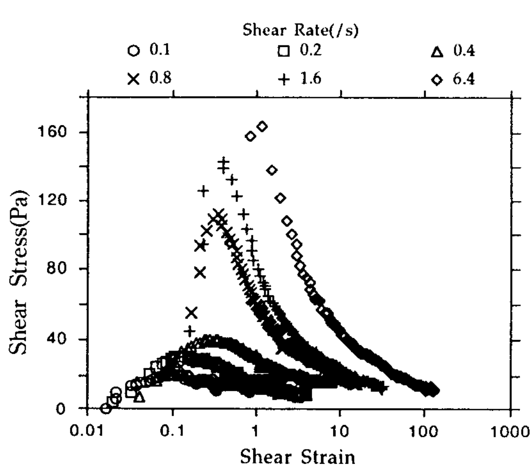


Fig. 1. Rheological behavior of cement paste under small shear rate.

cement paste has a yielding behavior and is sensitive to experimental conditions. At large deformation, however, cement paste has a broken structure and approximates a constant structure regardless of experimental conditions. Cement paste rheology was mainly studied at large deformation region; that is, interested in an equilibrium state after structure breakdown. At large deformation experimental conditions do not affect rheological properties of cement paste. However, the experimental conditions are important to analyze the microstructure of cement paste from the relationship between the interactive strength and the rheological properties of cement paste. The transition region between the structural build-up (elastic solid region) and the structural break down (non-Newtonian liquid region) can be quantified by the experiment with small deformation region. Yield stresses measured at small strain region are connected to the flocculation forces of cement particles based on attractive or repulsive forces between particles. In this paper the importance of experimental conditions will be discussed at the region of small deformation through the geometry effect of a sensor.

The ability to measure small strain behavior of cement paste is related closely to the type of sensor especially when the experiment is performed under relatively long time and quiescence. Reported rheological characteristics of cement paste were frequently inconsistent. There are many reasons for this including different mixing history [3-5], inhomogeneous characteristics of cement paste [6], and variations in experimental methods that include both the type of a sensor and the loading method [7]. Coaxial cylinder viscometers have been used a great deal as cone/plate and plate/plate viscometers.

Coaxial cylinder viscometers [8, 9] are easy to use and the shear history of cement paste is controllable. The shear rate throughout a specimen is uniform as long as the ratio between inner and outer diameters is close to 1(<1.10) i.e. the velocity profile across radius remains linear. Samples can be isolated from the atmosphere to avoid environmental effects. Disadvantage of coaxial cylinder viscometers includes problems with plug flow [10], slippage at the walls of cylinders [11, 12], sedimentation (vertical concentration gradient) [13], centrifugation (horizontal concentration gradient) and end effects [14].

Cone/plate viscometers [7, 15, 16] have also been used to measure the rheology of cement paste. Jones and Taylor [7] used a truncated cone/plate viscometer but the gap was not reported. They suggested that the difference between their results (anti-thixotropic) and results from coaxial cylinder viscometers (thixotropic) was attributable to the different geometries. In other words, the cone/plate viscometer has a constant shear rate throughout a specimen at a particular speed of rotation, but the coaxial cylinder viscometer had varying shear rate and associated shear stress.

A plate/plate viscometer has been used to carry out dynamic tests [17] with constant gap of 1 mm, and small strain test [2] with constant gap of 2 mm. The plate/plate viscometer has advantages over cone/plate viscometer because the gap between a sensor and a platen is uniformly much larger than the size of cement particles although cone/plate geometries can cause erroneous results due to particles binding in the small clearance near the axis. An additional advantage of this type of viscometer is that inertial effects can be accommodated very simply [18]. An important disadvantage of this geometry is that the strain in the sample is not uniform, but varies linearly from zero at the center to the maximum at the outer diameter. This presents difficulties in interpreting the measurements of non-linear materials.

Although there were advantages of the plate/plate and cone/plate geometries, they produced variable results and these have not been analyzed in detail. To increase the domain of the usage of plate/plate and cone/plate viscometers, artifacts are investigated in this paper. In an effort to establish an experimental technique using these geometries, the following factors were studied: i) the feasibility of using a truncated cone/plate sensor in cement paste rheology by considering the torque distribution, ii) problems associated with the amount of a sample and related edge effects, iii) the effect of sensor geometries such as the diameter and the size of gap (these factors were also related to the amount of a sample), and iv) the atmospheric condition, especially, the relative humidity around a sample.

2. Experimental Methods

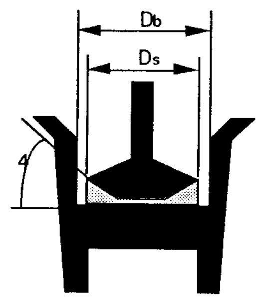
The experimental system [19] was operated with a viscometer capable of rotational and oscillatory control using plate/plate, cone/plate, and coaxial cylinder sensors; the measurement system was manufactured by HAAKE (Model RV20, RC20 and CV20N). Rotational experiments were performed to study the effect of sensor geometry. The measuring unit consists of two parts; a lower container which holds the sample and rotates at a controlled rate, and a upper sensor which is stationary and senses the torque to compute the average of shear stress. The specifications of various sensors used are given in Table 1.

A cone/plate sensor produces less ambiguous information than a plate/plate sensor because the material is deformed at a constant shear rate. However, because the gap between a sensor and a platen at the center is very small compared to the size of particles or flocs, it can not be applied to the study of cement paste rheology. The spacing between a sensor and a platen should be at least 10 times greater than the size of the largest particle [14]. In the case of cement paste, the gap size should be at least 0.7 mm since the maximum particle size is about 60~70 μm [1]. To avoid this problem in the experiment, the cone tip at the center was truncated to increase the gap size to 0.7 mm with the same cone angle as 0.0698rad (see Fig. 2). The influence of a truncated cone tip on torque is investigated under the assumption that a Newtonian liquid is placed between a sensor and a platen.

The truncated cone/plate sensor is composed of platen and cone parts. At a platen part, using cylindrical coordinates (r, θ, z), the velocity distributions are:

Table 1. Specifications of various types of sensor

Sensor	plate and plate		truncated cone and plate	
System	PQ20	PQ45	PK30	PK45
D_b (mm)	45	45	45	45
D_s (mm)	19.57	41.74	27.83	41.74
sample volume (cm^3)	.204, .582	.958, 2.737	0.324	1.11
gap size (mm)	.7, 2	.7, 2	.7 (non-truncated)	.7 (truncated)



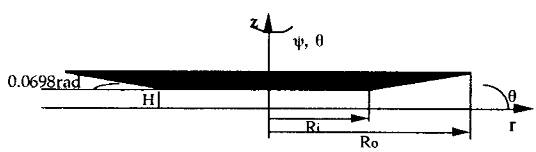


Fig. 2. Dimensions of a truncated cone/plate sensor.
cylindrical coordinate for plate-plate part: r, θ, z
spherical coordinate for cone-plate part: r, θ, Ψ
H: gap size(mm)
Ri: inner radius of sensor(mm)
Ro: outer radius of sensor(mm)

$$V_r=0$$
$$V_\theta=\frac{r\omega z}{H}$$
$$V_z=0 \tag{1}$$

The relationship between stress and velocities throughout the sample can be expressed as:

$$\tau_{\theta z}=\mu\left(\frac{1}{r}\frac{\partial V_z}{\partial\theta}+\frac{\partial V_\theta}{\partial z}\right) \tag{2}$$

where $\tau_{\theta z}$ is equal to $\tau_{z\theta}$ by symmetry and μ is a constant viscosity from the assumption of a sample as Newtonian liquid.

By inserting Eq. (1) into Eq. (2), Eq. (2) is:

$$\tau_{\theta z}=\mu\frac{r\omega}{H} \tag{3}$$

Then, the torque is obtained as follows:

$$T_{p\text{-}p}=\int\tau_{\theta z}\,rdA=2\pi\int_0^{R_i}r\frac{\mu r\omega}{H}rdr=\frac{\pi\omega\mu R_i^4}{2H} \tag{4}$$

At a cone part, using spherical coordinates (r, θ, ϕ), the velocity distributions are:

$$V_r=0$$
$$V_\theta=0$$
$$V_\phi=\frac{r\omega z}{z_o} \tag{5}$$

where $z=r\tan\theta$ at $0<\theta<\psi_o$
$z_o=r\tan\psi_o$ at $\theta=\psi_o$

Because ψ_o is very small in this sensor as 4° (=0.0698 rad), $z=r\theta$, $z_o=r\psi_o$. Then,

$$V_\phi=r\omega\frac{\theta}{\psi_o} \tag{6}$$

The relationship between stress and velocities in spherical coordinates is expressed by:

$$\tau_{\theta\phi}=\mu\left(\frac{\sin\theta}{r}\frac{\partial}{\partial\theta}\left(\frac{V_\phi}{\sin\theta}\right)+\frac{1}{r\sin\theta}\frac{\partial V_\phi}{\partial\phi}\right) \tag{7}$$

where $\tau_{\theta\phi}$ is equal to $\tau_{\phi\theta}$ by symmetry, and $\sin\theta\rightarrow\theta$ as a constant, then Eq. (7) can be rearranged:

Table 2. Torque distribution between cone and plate parts

H (mm)	R_i (mm)	small diameter (19.57 mm)		large diameter (41.74 mm)	
		$T_{p/p}/T$ (%)	$T_{c/p}/T$ (%)	$T_{p/p}/T$ (%)	$T_{c/p}/T$ (%)
0.175	2.50	0.80	99.20	0.25	99.75
0.35	5.01	3.53	96.47	1.04	98.96
0.7	10.02	30.8	69.20	8.50	91.50

$$\tau_{\theta\phi}=\mu\frac{\omega}{\psi_o} \tag{8}$$

The torque is obtained as follows:

$$T_{c\text{-}p}=\int\tau_{\theta\phi}\,rdA=2\pi\int_{R_i}^{R_o}r\frac{\mu\omega}{\psi_o}\,rdr=\frac{2\pi\mu\omega R_i}{3H}(R_o^3-R_i^3) \tag{9}$$

From Eqs. (4) and (9), the total torque is:

$$T=T_{p\text{-}p}+T_{c\text{-}p} \tag{10}$$

The distribution of torque between the horizontal platen and the slanting cone parts of the sensor is shown in Table 2 as a function of the diameter of the plate and, therefore, the corresponding distance between a truncated platen and a lower platen at the center. A large diameter sensor shows less change in torque distribution compared to that of a small diameter sensor. Interferences must be considered because a large diameter sensor has only a small spacing between it and the wall that bounds the specimen container. The results in Table 2 suggest that for Newtonian liquid, the truncated cone/plate sensor with large diameter can be considered as a non-truncated cone/plate sensor with an error in the range of 8.5% for torque.

A Type I portland cement was used to prepare cement paste. The ratio of water:cement by mass was fixed at 0.4. A paste was mixed by hand for 90 seconds; then, it was transferred into the lower container. When a specimen was placed between a sensor and a lower container an ideal boundary would be a frictionless vertical edge. Unfortunately, cement paste falls down due to its own weight and it will partially flow out from the gap between sensor and platen. If there is an extra amount of sample it will impinge on the wall of lower container. The influence of a redundant sample is an important experimental variable in this study. An interrupted shear rate experiment is used, and consists of three measurement stages of 120 seconds under a constant shear rate of 0.5 s^{-1} interplaced with three rest stages of 120 seconds as shown in Fig. 3. Experiments are performed to explore the effect of artifacts on structural rebuild-up as well as on structural breakdown of a cement paste.

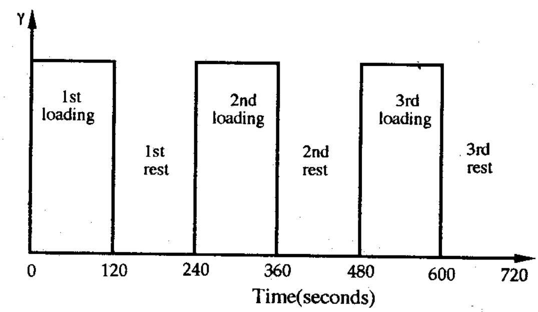


Fig. 3. Step function in shear rate experiment.

3. Experimental Design and Results

3.1. Effect of the Amount of a Sample on the Shear Stress Distribution

The plate/plate sensor with a diameter of 19.57 mm was used to contain various amounts of a sample. A dimensionless diameter was defined as the ratio of the diameter of a specimen divided by the diameter of a upper sensor after finishing the experiment i.e. it means the extra amount of sample. Six kinds of sample amounts were used, each defined as a dimensionless diameter by the 1, 1.158, 1.368, 1.579, 1.895, and 2.21. Fig. 4 shows

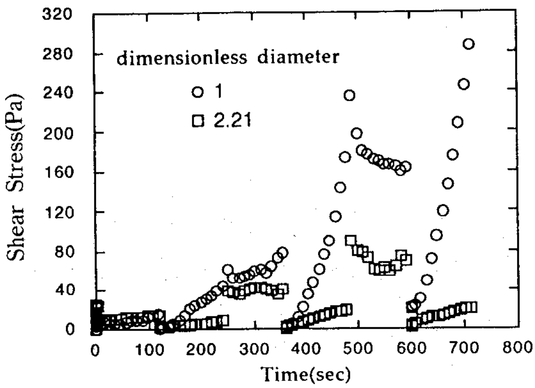


Fig. 4. Shear stress vs. time for different amount of sample.

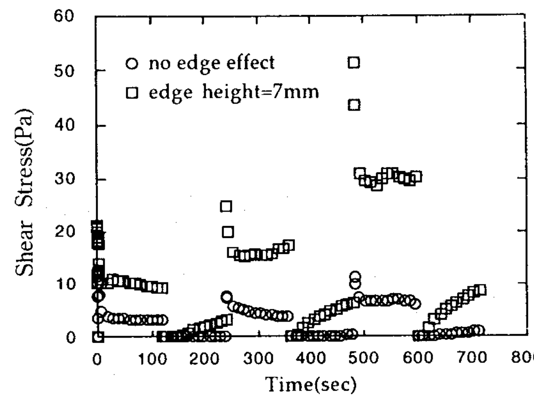


Fig. 5. Shear stress vs. time for various amount of sample.

the relationship between shear stress and time for two samples with dimensionless diameters of 1 and 2.21. The amount of a sample affected yield and end stresses during either deformation or rest periods. Here, the yield stress is defined as the peak stress occurring as soon as the experiment begins, and the end stress is defined as the stress at the end of deformation or rest periods. The influence of sample amounts may be effectively analyzed by investigating the yield and end stresses because the yield stress represents the state of an undamaged structure at the deformation period or the state of a recovered structure at the rest period, and end stress represents the effect of a shear rate at deformation period and the effect of a stationary state at rest period. There is no mechanical reason why the amount of a sample should influence the measured results. The build up of structure as a result of chemical reactions should not be affected either by the amount of a sample. However the exposed surface of a sample is a function of the amount of a sample, and the possibility that surface drying causes artifacts may be explained. This shows that the evaporation from the surface of a sample can affect the inside structure of a sample.

Edge effect comes from a small gap between a sensor and the wall of a bottom container if a large diameter sensor is used. The truncated cone/plate sensor with a diameter of 41.74 mm was used to contain various amounts of sample. The edge height at the wall of a bottom container is defined as the height of a squeezed sample by a upper sensor due to a redundant sample compared to the volume between a upper sensor and a bottom platen. Edge heights were varied to be the 0, 1, 3, 4, and 7 mm by controlling the amount of a sample. Fig. 5 shows the relationship between shear stress and time for two kinds of edge heights. The increase of the yield and end stresses with increasing an edge height is purely accredited to the effect of edge height because the evaporation of a sample is already precluded by a large diameter sensor. The end stress measured in the rest period is small compared with the yield and end stresses measured during deformation period. This means that cement paste is prevented from drying when using a large diameter sensor. From the comparison of the results between small and large diameter sensors, the artifact due to drying at a small diameter sensor is greater than that of a large diameter sensor.

3.2. Effects of Diameter and Gap Size of Sensor on the Shear Stress Distribution

Even though the sensor dimensions such as diameter and gap size, are related to the amount of a sample as discussed in the previous section, experiments which preclude the influence of a redundant sample are performed to investigate the following: (i) the effect of a gap size holding the same diameter, (ii) the effect of a diameter hol-

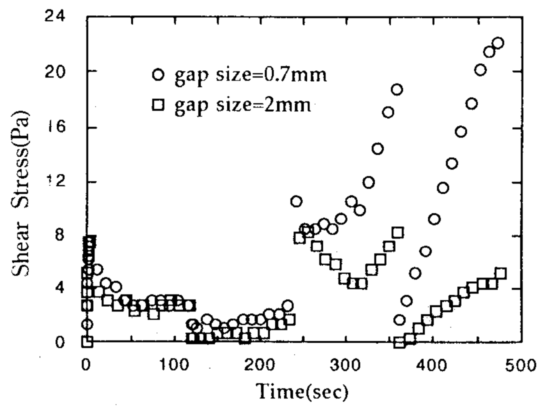


Fig. 6. Effect of gap size under same diameter of sensor (D_S=19.57 mm).

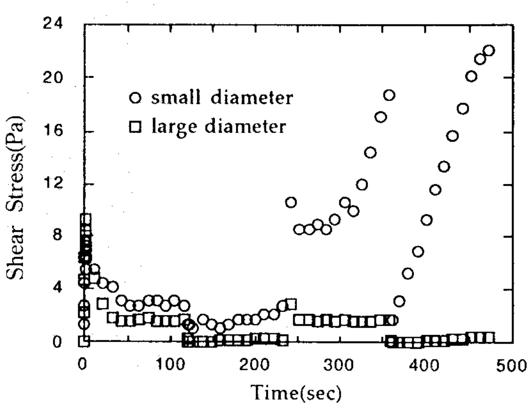


Fig. 7. Effect of diameter of sensor under same gap size (gap size=0.7 mm, small diameter: PQ 20, and large diameter: PK45).

ding the same gap size.

In case (i), as shown in Fig. 6, there are not many differences between results regardless of gap size during the 1st deformation and rest periods, but after these periods, the lower the gap size, the higher the shear stress is. It does not mean that this is an intrinsic behavior of cement paste. However, it may come from the different degrees of drying of the sample due to different quantities of a sample used. In case (ii), the behavior of a sample is similar at initial stage regardless of diameters of a sensor as shown in Fig. 7. At later stage, however, the smaller the diameter of a sensor, the higher the shear stress is. These differences are related to the large surface-to-volume ratio of a sample exposed to the atmosphere for a small diameter sensor. In the case of a truncated cone/plate sensor, the structure breakdown curve is smooth compared with that of a plate/plate sensor. This may be because the former has a constant shear rate across its radius, but the latter has a variable shear rate. The influence of a sensor geometry on the shear stress becomes large with decreasing gap size or sensor diameter.

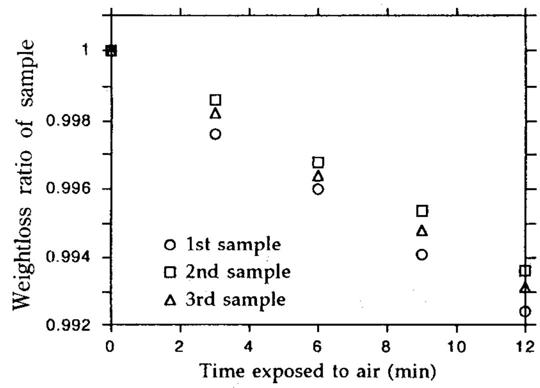


Fig. 8. Weight loss of cement paste exposed to air (RH=40%).

3.3. Effect of Relative Humidity

All of the above experiments were performed with a sample partly exposed to air. Fig. 8 shows the weight loss ratio of a sample exposed to air (RH=40%). The weight of a sample was measured every 3 minutes using an accurate balance with four digits and was divided by an initial sample weight. Experiments were repeated three times to check reproducibility. There was about 0.7 % weight loss in 12 minutes. A new water:cement ratio after 12 minutes may be calculated as 0.3976 because the water is evaporated as much as 0.7% of the initial sample weight. There is little change in the water:cement ratio between the beginning and the ending of the experiment. As already argued, drying may influence the results even though only the part of a sample is exposed. This can be avoided by preventing a sample from drying using a wetted cup, in which wetted sponge or paper is pasted as shown in Fig. 9. Fig. 10 shows the drying effect on the sample which is

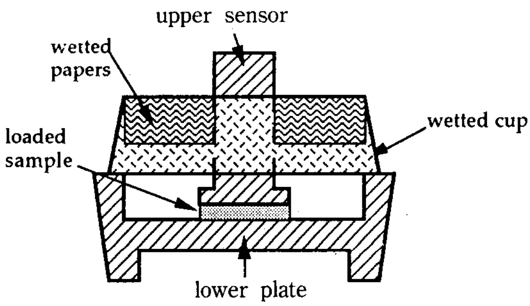


Fig. 9. Usage of wetted cup.

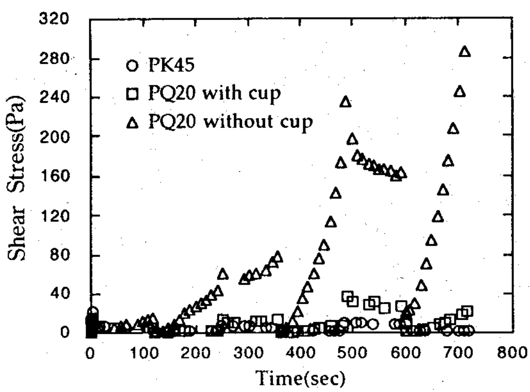


Fig. 10. Effect of environmental condition around of sample on shear stress.

deformed under both a plate/plate sensor (PQ20 in Table 1) and a truncated cone/plate sensor (PK 45 in Table 1) with the same gap size (0.7 mm). When the relative humidity around a sensor is 40%, the sensor with a small diameter had larger influence on drying of a sample than the sensor of a large diameter. Because of the relatively short period, the idea that evaporation could influence the results was not obvious. However, when a wetted cup was used, the results of the two types of sensors became much closer and they were reproducible. Even during the short duration drying causes substantial stiffening of at least part of a specimen. This is an effect that is not accounted for in the literature and, apparently, it must be controlled if accurate measurements of rheology of cement paste are to be made.

4. Conclusions

A truncated cone/plate sensor, that was made by increasing the gap between cone tip and bottom platen, had advantages over plate/plate sensors. Most of the sample deformed uniformly and the binding effect caused by particles trapped at the cone tip was eliminated. Both the diameter of a sensor and the gap size between a sensor and a bottom platen were varied to study the effect of a sensor geometry. Artifacts due to the evaporation of a water from a sample have been correlated to various sensor geometries. When the evaporation from a sample is prevented, the rheological characteristics of cement paste can be measured with a reproducibility not possible in the past. This is a new and important consideration when measuring the rheology of cement paste.

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