POF based smart sensor for studying the setting dynamics of cement paste

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Abstract. Fiber optic smart sensors are used to monitor the civil structures. One of the important parameters in civil engineering is the setting characteristics of concrete made of cement. The paper discusses how a simple polymer optical fiber can be used to characterise the setting dynamics of various grades of cement. The results explain the comparative performance of polymer fiber over silica fiber. The basic principle underlying the sensor is that as the cement sets, it exerts a stress on the sensing fiber, which is laid within the cement paste. This stress induces strain on the optical fiber, which can be thought of as a series of aperiodic microbends on the surface of the fiber. This in turn changes the characteristics of the light signal transmitted through the fiber and can be viewed as stress induced modulation of light in the fiber. By monitoring the intensity variation of transmitted light signal with time we can determine the cement setting rate. This can be used as an effective tool for quality testing of commercially available cements of different grades.

Over the last century, concrete [1, 2] has changed the way we dwell on this planet. It has been established as the most popular building material with unmatched properties. It is such a familiar material that quite often we ignore the remarkable process by which cement and water are mixed with a wide range of aggregates to form a plastic mass which ultimately sets into a strong and durable material.

Moisture or water plays an important role in the setting and strength development of concrete. The cement hardening is due to the chemical process called hydration. This means that the silicate and aluminate [3] minerals in the cement react and combine with water to produce the ‘glue’ that holds together the aggregate which forms the concrete. The water-cement ratio is always much in excess of that required for hydration of the cement in the attainment of its final form in the concrete. The excess water used for workability will be lost as the cement gradually attains its final form. The cement paste has several other important properties such as strength and porosity, which are determined by the ratio of water to cement in the original mix. The water-cement ratio is an index of strength and design of cement mixes. The lower the water cement ratio, the higher will be the strength of the hardened paste and that of the concrete.

Many studies have been done in the past for the optimization of cement paste used for civil structures [4, 5, 6]. Conventional technique for the measurement of setting time of cement mix is by using the Vicat and Gillmore needles [1, 2]. These measurements can provide only the initial and final
setting time. Since the characteristics may change from batch to batch of the same grade of cement it is very important to study the setting behavior of the cement paste. There are many chemical and physical reactions taking place during the setting of concrete, which are not fully understood till now. This is due to the fact that wide ranges of chemical substances exist in the cement. The chemical reactions may continue slowly over a long period of time and others may be initiated by various environmental parameters into which concrete is subsequently exposed. For these studies, continuous measurement of these setting and curing characteristics of cement are very important. Conventional methods do not provide continuous measurement of setting characteristics. But with the present fiber optic sensor we can continuously monitor setting and hardening pattern of different grades of cement.

Optical fiber sensors are capable of playing an important role in the health monitoring of civil structures such as bridges [7], dams, [8] buildings and so on. The main features of the optical fiber sensor are its immunity from electromagnetic noise, very good sensitivity and compactness [9]. Since optical fibers are very sensitive to strain and bending losses, a variety of intensity modulated sensors can be used to carry out in-situ studies of civil structures [10]. Studies have been carried out to determine the cracks in concrete structures using displacement techniques [11]. Moreover, many interferometric sensors based on Bragg gratings have been developed for more sensitive measurements, such as vibrations and strain [12, 13]. Distributed fiber optic sensors can also be used as smart sensors for the determination of cracks in concrete structures [14, 15].

The optical fiber sensor can be incorporated into such structures during their construction or can be adhered to them after the completion of construction. The sensitivity of the sensor largely depends on the bonding properties of the construction materials [2].

The main advantage of the present method is that no mechanical parts are involved in the measurements, and this reduces the instrumental error to a great extent. Moreover, the experimental setup is very simple and straightforward and this can be installed at the work site with no sophisticated instruments for measurements. Commonly available high luminescent LEDs and photodiodes can be employed for these studies, which make the sensor very cost effective. The use of plastic optical fiber will further reduce the cost considerably.

1. Setting and hardening of cement paste

Setting is described as the stiffening of the cement paste, i.e. setting refers to a transition from fluid to a rigid state. In current practice the terms ‘initial setting’ and ‘final setting’ are used to describe the stages of setting, which take place within ten hours. The setting process is accompanied by temperature changes in cement paste. Initial set corresponds to a rapid rise in temperature and final set to a peak in temperature. The usual method for finding the setting time of cement is by Vicat apparatus. Usually a measure of the above said two setting times, viz the initial and the final setting times follow a definite procedure. The period elapsing between the time when water is added to the cement and the time at which the Vicat needle fails to pierce the test block by 5± 0.5mm is taken as the initial setting time [2].

The period elapsing between the time when water is added to the cement and the time at which the needle makes an impression on the surface of the test block while the attachment fails to do so is taken as final setting time[2]. Typically, the initial setting value for ordinary porcelain cement should be higher than 30 minutes while final setting time should be less than 600 minutes [2].

This means that in the usual description, the setting of the cement will be completed within 600 minutes. However, the dynamics of cement setting continues for duration of more than 600 minutes, the characteristics of which cannot be studied using the Vicat apparatus. This is one of the important drawbacks of the Vicat-based measurements. Since the Vicat measurements provide only the
information about the setting times within 600 minutes, the curing and rapidity with which cement achieves hardness with different environmental conditions cannot be studied. Although, during the setting process, the paste acquires some strength, for practical purposes, it is the hardening, which indicates the gain of strength of set cement paste. The speed of setting and rapidity of hardening are entirely independent of one another. Using conventional techniques, the speed of setting and hardening cannot be traced. The fiber optic based measurements will be helpful to study the complete setting characteristics of the cement paste.

2. **Principle**

The basic principle underlying the sensor is that as the cement sets, it exerts a stress on the sensing fiber, which is laid within the cement paste. This stress induces strain on the optical fiber, which can be thought of as a series of aperiodic microbends on the surface of the fiber.

![Figure 1: Stress induced microbends in an optical fiber](image)

This in turn changes the characteristics of the light signal transmitted through the fiber and can be viewed as stress induced modulation of light in the fiber. By monitoring the intensity variation with time we can accurately determine the cement setting rate. This can be used as an effective tool for quality testing of commercially available cements of different grades. Microbends on the fiber arise as the variable pressure exerted on it distorts the fiber [16, 17, 18]. These bends are so small that the bend radii are of the order of the diameter of the fiber. If the mechanical perturbation is severe, a major part of the light is coupled to the cladding and is lost as radiation modes. Essentially, a continuous succession of such small bends may cause a significant enhancement of attenuation in the fiber (figure 1). The small variations in the core diameter due to stress induced deformation on the fiber during the setting of the cement mix, give rise to a scattering mechanism, which accounts for part of the loss. The microbends formed cause the coupling of energy between various guided modes and leaky modes (both cladding and radiation modes), the latter giving rise to a loss during transmission of light.

3. **Experimental setup**

The experimental set-up essentially consists of a super luminescent LED source, a certain length of multimode uncladded silica fiber or a polymer optical fiber (PMMA) and a photo detector. The epoxy lens of the commercially available LED (\(\lambda=670\) nm.) is removed and the end face is thoroughly polished, so as to make a very efficient coupling to the fiber. The experimental setup is as shown in figure 2.
Initially the sheath and the cladding in the sensing region of the fiber are removed. This ensures the development of micro-bends on the fiber due to local strain. The ends of the fiber are polished to obtain efficient coupling from the LED and to the power meter.

The fiber is placed in such a manner that its uncladded sensing region is well within the container. A cement mixture with a specific water cement ratio (cement: water is 4:1) is made and it is poured into the container (50mm x 50mm x 20mm) such that it fully covers the sensing region. Only a small quantity of the cement mix is required for this purpose. The output power measurements are taken at regular intervals of time using a Newport 1815C power meter. Data acquisition and processing are done using a Labview card and a PC.

4. Results and discussions

The output variation in transmitted power through the laid fiber with time for 43 grade cement mix is studied. Typical plots of power vs setting time for glass fibers are shown in figures 3a & 3b and for plastic fibers in figures 4.a & 4.b. As can be clearly seen from the figures, the dynamics of the setting of cement mix involves three phases, viz., an initial slow phase, the second rapid phase and the final slow setting phase [2].

Two sets of experiments were carried out, one without hydrating and the other by hydrating at regular intervals. It can be clearly seen from the graphs that by using polymer optical fiber as the sensing element the sensitivity are increased to a greater extent. Moreover, the initial setting region which had a very low sensitivity in the first setting phase by using glass fibers has been enhanced to a greater extent when polymer optical fibers were used. One can clearly see that the initial setting phase also has a gradual setting pattern. But this was not evident from the experiments based on glass fiber. In the second set of experiments the cement mix was allowed to set and was hydrated at regular intervals.

During the initial phase, the cement loses its plasticity and becomes a thick paste. Here, we can infer that the cement mix starts the process of setting. During this phase, the cement passes through the initial and final setting processes as described in literature [1, 2]. Then it enters the second phase, which is the most important and the fastest process in the whole phenomenon of setting of cement. In summary, by evaluating the setting properties of the second stage one can determine whether the sample in question is showing the properties of the required grade. Thus for verifying the data provided by the manufactures, we can determine the setting times and hence the quality of the cement samples. Observation during the third and final setting phase reveals that the cement mix attains its full strength in almost a month’s time. As mentioned before, the results from the Vicat experiment provides the details of the initial and final setting times. However, the results from the fiber optic
smart sensor show not only the setting times given by Vicat apparatus but also the setting characteristics extending upto a few weeks. The so-called initial and final setting times are 160 and 420 minutes respectively for 43-grade cement, which agrees with the Vicat test. The differences in setting behavior of cement mix have far reaching implications in civil works related to building constructions. A carefully incorporated fiber optic sensor during building construction will be helpful in identifying proper phases of setting.

**Figure 3a, b:** Plot showing the output optical intensity vs. time: 43 grade cement was used for the study.
4.1 Modification of porosity during cement setting

To study the modification of porosity in a concrete mix during the setting period, water is added at different setting stages of the curing process. This causes discontinuities in the curing curve, as is evident from the graphs 3.b and 4.b. The amount of shift in the discontinuity on the curing curve directly reflects the reduction in the number of micropores in the cement paste as the setting progresses. To study the setting process continuously the experimental setup was interfaced to a computer with the help of LabView software.

When water is added at specific times, the water percolates through the micropores onto the fiber laid inside the cement paste. Due to the presence of water instead of air, the cladding modes will be guided back into the fiber, thus increasing the output optical power. Setting of cement mix involves three phases, viz., an initial slow phase, the second rapid phase and the final slow setting phase [2]. During the initial phase, the cement loses its plasticity and becomes a thick paste. Here, we can infer
that the cement mix starts the process of setting. During this phase, the cement passes through the initial and final setting processes as described in literature [1, 2]. Then it enters the second phase, which is the most important and the fastest process in the whole phenomenon of setting of cement. Conventional technique followed by civil engineers provide information regarding the dynamics of cement setting during first phase, where both initial and final set completes.

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