Variation of Physical Properties of Cement over Time

Riazul Haque, Sumit Purkayastha, and Abdul Muktadir Khan

***Abstract*—Cement, a binding material, is considered the key ingredient of concrete which is used in construction. Physical properties of cement indicate the quality of cement. But these properties change after a certain period, which may lead to unsuitability of use in construction. In the experiment, physical properties of cement like normal consistency, setting times, fineness, soundness and compressive strength have been determined following ASTM (American Society for Testing and Materials) & BDS EN (Bangladesh Standards Engineering Division) standards. Experiment has been carried out for two types of cement sample- PCC (Portland Composite Cement) 42.5N and OPC (Ordinary Portland Cement) 52.2N, using two standards on the same day at 0th, 4th & 8th week from the manufacturing date of cement. Maintaining a controlled environment of humidity and temperature in the laboratory, change of physical properties over time has been analyzed from November, 2016 to January, 2017. The experimental result shows that the cement remains perfect for use till 8th week (approximately 2 months) from the manufacturing date and the values of physical properties remain within the specified range of ASTM & BDS EN standards; except the compressive strength of PCC 42.5N, which is lower than that of BDS EN standard.**

***Index Terms*—ASTM and BDS EN standards, compressive strength, OPC 52.5N, PCC 42.5N, physical properties.**

I. Introduction

As construction industry of Bangladesh is developing day by day, the demand of cement is increasing rapidly. So cement industry is booming in the country. But there is a problem in the use of cement after a certain period of time because the variation of physical properties over time deteriorates the quality of cement.

This experimental program focuses the variation of several physical properties like Normal Consistency, Fineness, Compressive Strength, Initial and Final Setting Time etc. over time showing comparison of two major types of cement.

II. Experimental Program

The whole experimental program was carried out in the laboratory to determine normal consistency, initial and final setting time, compressive strength, fineness and soundness of OPC 52.2 N and PCC 42.5 N at 0th, 4th and 8th week from the manufacturing date of cement following ASTM & BDS EN standards. The laboratory in which specimens were prepared and tested, maintained a temperature of 20±2 ℃. The relative humidity of the laboratory was maintained above 50%. When it was required to prepare cement paste, the temperature of the mixing water was kept 23 °±1.7 ℃.

Reference [1] Normal consistency test was conducted according to ASTM standard requirements of specification C187-11 and BDS EN standard requirements of specification EN 196-3:2005(E). Vicat apparatus was used to determine normal consistency in both ASTM & BDS EN standards. Reference [2] Initial setting time and final setting time conformed to the ASTM standard requirements of specification C191-08 and BDS EN standard requirements of specification EN 196-3:2005(E). Vicat apparatus was used to determine initial setting time and final setting time in both ASTM & BDS EN standards. Reference [3] For compressive strength, test method conformed to the ASTM standards requirements of specification C109-11b and BDS EN standard requirements of specification EN196-1:2005(E). For ASTM standards, 27 two- inch cube specimens and for BDS EN standards 27 prismatic specimens of 160mm×40mm×40mm wereused for compressive strength test. Reference [4] Fineness test was performed in accordance to ASTM standard requirements of specification C204-11. Blaine air-permeability apparatus was used in fineness test. Reference [5] Soundness test was performed in accordance to ASTM standards requirements of specification C1038-01. The effective gage length of specimens was 250 mm. in soundness test.

A. Normal Consistency

Reference [6] the ring was placed containing cement paste and the glass plate in the specified location of the Vicat apparatus and brought down the flat end of the plunger so that it may just touch the paste surface and tighten the set-screw. The movable indicator was set to the upper zero mark of the scale or takes the initial reading. The rod was released immediately and allowed penetrating into the cement for 30 sec and the scale was read after 30 sec (i.e. the depth of penetration). Normal consistency was obtained when the plunger settles 10±1 mm below the surface of the cement paste in 30 sec. The entire operation was repeated using different water percentages and fresh dry cement until the desired value of consistency was reached. Fig. 1 shows the Consistency test in the lab.

The amount of water required was calculated for normal consistency to the nearest 0.5% of the weight of the dry cement.

B. Initial Setting Time and Final Setting Time

Reference [7] The cement paste sample was kept in the moist cabinet or moist room undisturbed for 30 mins after molding. After 30 mins, the first penetration test was made. The tip of the needle was lowered to touch the surface of the paste. The set screw was tightened and the indicator was set at the upper end of the scale or an initial reading was taken.

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The rod was released immediately and allowed to penetrate into the cement for 30 secs and the scale was read after 30 secs. The procedure of determining penetration was repeated every 15 mins until a penetration of 25 mm or less was obtained. Each penetration is was made at least 5 mm. away from any previous penetration and at least10 mm. away from the mold. The results of all penetration tests were recorded and by penetration, the time was determined when a penetration of 25 mm. was obtained. The elapsed time between the initial contact of cement and water and the penetration of 25 mm. is the Vicat time of setting or Vicat initial time of setting. The final time of setting is the elapsed time between the time of initial contact of cement and water and the time when the needle does not sink visibly into the paste. Fig. 2 shows Mixture Machine for Setting Time Test.

Fig. 1. Consistency test in the BUET lab.

Fig. 2. Mixture machine for setting time test in BUET laboratory.

The time required for 25 mm. penetration is calculated from graph and expressed it to the nearest 1 min. This time is arbitrarily defined as initial setting time. The time when the needle does not sink visibly into the paste is the final setting time which is calculated to nearest 15 mins.

C. *Compressive Strength*

Reference [7] The specimens were tested immediately after their removal from storage water (specimens for 1-day test were brought from the moist closet or moist room). All specimens for a given test age must be broken within the permissible tolerance. If more than one specimen at a time was removed from the storage water for testing, these specimens are kept completely immersed in water at a temperature of 23 ± 2 ℃ (73.4 ± 3 ℉) until time of testing, each specimen was wiped to a surface-dry condition and removed any loose sand grains or incrustations from the cube faces. The critical cross-sectional dimensions were measured to the nearest 0.01inch. The load was applied to specimen faces that were in contact with the true plane surfaces of the mold. Carefully the specimen was placed in the testing machine below the center of the upper bearing block. No cushioning or bedding materials were used. The spherically seated block was brought into uniform contact with the surface of the specimen. An initial load was applied at any convenient rate up to about one-half of the expected maximum load. Thereafter the load was applied, so that the maximum load will be reached within next 20 to 30 seconds. No adjustments were made in the controls of the testing machine, while a specimen was yielding prior to failure. The maximum load indicated by the testing machine was recorded. Fig. 3 shows the temping for compaction, Fig. 4 shows curing of test specimens and Fig. 5 shows the Loading Machine.

Using the calibration equation of the strength testing machine the actual maximum load for the observed load is calculated. If the cross sectional area of a specimen varies more than 1.5 % from the nominal, the actual area is used for the calculation of the compressive strength. The average compressive strength of all acceptable test specimens made from the same cement and tested at the same period is determined nearest to 0.1 MPa.

Fig. 3. Temping for compaction.

Fig. 4. Curing of test specimens in BUET laboratory.

Fig. 5. Loading machine.

D. Fineness

Reference [7] The permeability cell was attached to the manometer tube ensuring an air-tightconnection (a little stop-cock grease was used to ensure an air-tight connection) and taking care not to disturb the prepared bed of cement. Slowly the air was evacuated in the right arm of the manometer using the pressure bulb until the liquid reached to the mark and then the manometer valve was closed. The liquid started to lower slowly because of air-flow through the cement sample into the manometer. The timer was started when the bottom of the meniscus of the liquid reached the second mark from the top and the timer was stopped when the bottom of the meniscus reached the third mark (next to the bottom mark). This time was recorded to the nearest 1 second. Fig. 6 shows Blaine Air-permeability Apparatus.

Fig. 6. Blaine air-permeability apparatus in BUET laboratory.

For Portland cements and Portland cement-based materials, results are reported on a single determination on a single bed. The fineness of the cement is reported to the nearest 1 m^2/kg.

E. Soundness

Initial storage: Reference [7] Immediately upon completion of molding, the test specimens were placed in the moist closet or moist room for 22.5±0.5 hrs with their upper surfaces exposed to the moist air but protected from getting wet through water. After this time, the specimens were removed from the molds, properly identified and placed in water maintained at 23±2.0 ℃ for at least 30 mins prior to making the initial measurement.

Subsequent Storage: Reference [7] After the initial measurement, the test specimens were stored in saturated lime water in a tank in the moist cabinet or moist room. The specimens were kept with at least 5-mm (1/4-inch) of water during storage. Storage tanks were used which are constructed on non-corroding materials. The storage water was kept clean by changing as necessary.

Length Change Measurement: Reference [7] The specimens were removed from water storage, one at a time and only the gage studs were wiped with a damp cloth before measuring. Length-comparator readings were taken. The effective gage length of specimens was 250 mm. or 10 inch for the standard mold size. The first reading was made at the age of 24 hrs±15 mins from the time the cement and water were mixed together. Additional length-comparator readings were made at the age of 14 days. Fig. 7 shows Test specimens.

The change in length of the specimen is calculated at 24 hrs and at 14 days to the nearest 0.001% of the effective gage length and reported as the expansion of the specimen at that period. The average length of change of the two specimens is reported to the nearest 0.001%.

Fig. 7. Test specimens.

III. Result and Discussion

A. Normal Consistency

TABLE I: Normal Consistency (%) of Cement at 0^{th}, 4^{th} and 8^{th} Week

Week	OPC(ASTM)	PCC(ASTM)	OPC(BDS EN)	PCC(BDS EN)
0^{th}	26.5	26	31	30
4^{th}	26.5	26	31	30
8^{th}	26.5	25.5	31.5	30

Table I indicates the variation of the values of Normal Consistency in percentage of PCC and OPC with storage time. Both ASTM and BDS EN standards are used. From TABLE I, The normal consistency of PCC 42.5N in ASTM standard is decreased by 1.9% at the 8^{th} week and of OPC 52.5N in BDS EN standard is increased 1.6% at the 8^{th} week. Other values are remained the same at 0^{th}, 4^{th} & 8^{th} week for both PCC 42.5N & OPC 52.5N in ASTM & BDS EN standards. All the values satisfy the required standard value specified in ASTM and BDS EN, except the normal consistency value in ASTM standard of PCC 42.5N at the 8^{th} week, which is 0.5% less from the required value. Fig. 8 shows Normal Consistency (%) of cement at different period. It summarizes the results shown in TABLE I. It indicates that, Normal Consistency (%) is almost the same for both PCC and OPC in ASTM and BDS EN standards. But in BDS EN standards, the Normal Consistency increase by a few percentages for OPC by the 8^{th} week. These experimental results show that normal consistency of cement didn't vary significantly until 8^{th} week from the production date of cement, because the quality of cement was good during that period.

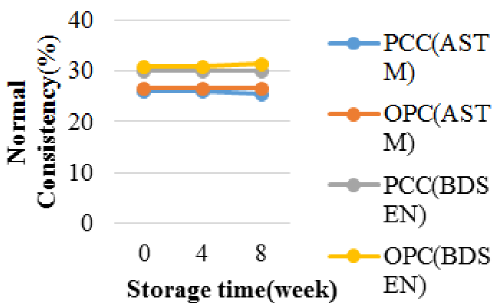

Fig. 8. Normal consistency (%) of cement at different periods (week).

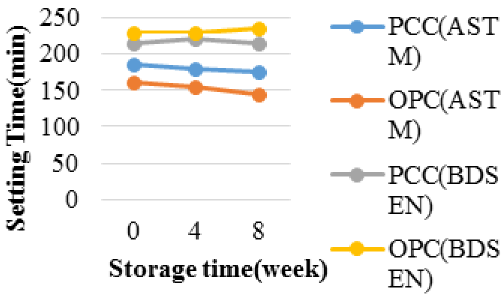

Fig. 9. Initial setting time (min.) of cement at different periods.

B. Setting Time

TABLE II: INITIAL SETTING TIME (MIN) OF CEMENT AT 0TH, 4TH AND 8TH WEEK

	Week	PCC (ASM)	OPC (ASTM)	PCC (BDS EN)	OPC BDS EN)
Initial Setting Time	0th	185	160	215	230
	4th	180	155	220	230
	8th	175	145	215	235
Final Setting time	0th	375	365	315	320
	4th	380	355	350	330
	8th	330	320	325	340

Table II shows the variation of both the initial setting time and final setting time in minute of PCC and OPC in ASTM and BDS EN standard by the 0th, 4th and 8th weeks. Initial setting time in ASTM standard for PCC 42.5 cement is decreased 2.7% at 4th week and 5.4% at 8th week. For OPC 52.5N cement it is decreased 3.1% at the 4th week and 9.4% at the 8th week. Besides, in BDS EN standard for OPC 52.5N cement initial setting time is increased 2.17% at the 8th week and for PCC 42.5N cement, initial setting time is increased 2.3% at the 4th week. All the values satisfy the required standard value specified in ASTM, BDS EN. Fig. 9 shows comparison of Final Setting Time (min) of cement at different period. It clearly indicates that, initial setting time of PCC cement is quite higher than OPC cement in ASTM standards but lower in BDS EN standards. So, it can be said that setting time can also be influenced by the standard used.

Final setting time in BDS EN standard for PCC 42.5 is increased 1.6% at the 4th week and 3.17% at the 8th week. For OPC 52.5N it is increased 3.1% at the 4th week and 6.25% at the 8th week. Besides, in ASTM standard for OPC 52.5N cement, final setting time is decreased 2.7% at the 4th week and 12.3% at 8th week. For PCC 42.5N cement, final setting time is increased by 1.3% at the 4th week but decreased by 12% at the 8th week. All the values satisfy the required standard value specified in ASTM and BDS EN. Fig. 10 shows Comparison of Final Setting Time (min.) of cement at different period of storage. It indicates that final setting time (min.) is higher for PCC than OPC in ASTM standards and almost the same for BDS EN standards. These experimental results show that Initial and Final setting time of cement didn't vary significantly until 8th week from the production date of cement, because the quality of cement was good during that period.

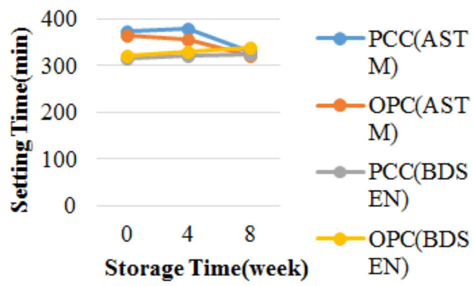

Fig. 10. Final setting time (min.) of cement at different period.

A. Fineness

TABLE III: FINENESS (M^2/KG) OF CEMENT AT DIFFERENT PERIOD OF STORAGE

Week	PCC(ASTM)	OPC(ASTM)
0th	474	439
4th	451	463
8th	468	447

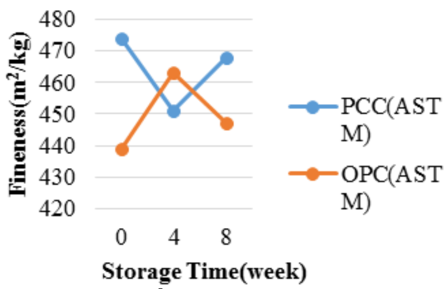

Fig. 11. Fineness (m^2/kg) of cement at different period.

Table III shows the variation of Fineness with the storage period. Fineness of cement in ASTM standard for PCC 42.5N is decreased by 4.85% at the 4th week and 1.3% at the 8th week. For OPC 52.5N, fineness is 5.5% increased by the 4th week and 1.8% by the 8th week. All the values satisfy the required standard value specified in ASTM. Fig. 11 shows Fineness (m^2/kg) of cement at different periods.From Fig. 11, it is evident that at first the Fineness of OPC is lower than PCC, but at the 4th week, it is greater for OPC. Then at the 8th week, again it decreases and becomes lower than PCC. These experimental results show Fineness of cement didn't vary significantly until 8th week from the production date of cement, because the quality of cement was good during that period.

B. Soundness

TABLE IV: SOUNDNESS (%) OF CEMENT AT DIFFERENT PERIOD OF STORAGE

Week	PCC(ASTM)	OPC(ASTM)
0th	0.294	0.428
4th	0.262	0.33
8th	0.24	0.288

Table IV is constructed to show the variation of Soundness with the storage time. Soundness in ASTM standard PCC 42.5 cement is decreased 10.88% at the 4th week and 18.37% at the 8th week. For OPC 52.5N cement it is decreased 22.9% at the 4th week and 32.7% at 8th week. All the values satisfy the required standard value specified in ASTM. So, it is clear that Soundness of both the types of cement is decreased with longer storage period. Fig. 12 shows Soundness (%) of cement at different period and also proves the result shown in TABLE IV. These experimental results show that Soundness of cement didn't vary significantly until 8th week from the production date of cement, because the quality of cement was good during that period.

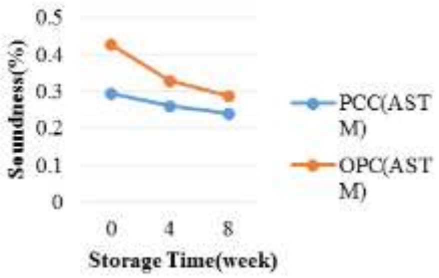

Fig. 12. Soundness (%) of cement at 0th, 4th and 8th week.

C. Compressive Strength

TABLE V: COMPRESSIVE STRENGTH (MPA) OF CEMENT AT DIFFERENT PERIOD OFSTORAGE

		0th week		4th week		8th week	
Standard	Days	PCC	OPC	PCC	OPC	PCC	OPC
ASTM	3	23.21	36.00	19.38	31.78	21.14	34.72
	7	31.03	43.90	28.21	40.83	27.98	40.29
	28	38.24	53.64	36.82	49.22	38.77	52.48
BDS EN	2	19.76	32.46	16.63	29.95	16.33	23.57
	7	35.21	45.39	29.81	40.09	28.25	38.82
	28	48.02	58.46	43.03	57.68	41.92	62.16

Table V shows the variation of Compressive Strength (MPa) in different stages of storage.

2 Days Compressive strength: 2 days compressive strength in BDS EN standard for PCC 42.5N is decreased 15.84% at the 4th week and 17.4% at the 4th week. For OPC 52.5N, it is decreased 7.7% at the 4th week and 27.38% at the 8th week. All the values satisfy the required standard value specified in BDS EN. Fig. 13 shows 2 Days Compressive strength (MPa) of cement by BDS EN method at different period.

3 Days Compressive strength: 3 days compressive strength in ASTM standard for OPC 52.5N is decreased 11.7% at the 4th week and 3.56% at the 8th week. For PCC 42.5N cement it is decreased 16.5% at the 4th and 8.9% at the 4th week. All the values satisfy the required standard value specified in ASTM. Fig. 14 shows 3 Days Compressive strength (MPa) of cement by ASTM method at different period.

7 Days Compressive strength: 7 days compressive strength in ASTM standards for PCC 42.5N is decreased 9.1% at the 4th week and 9.8% at 8th week. For OPC 52.5N cement it is decreased 7.5% at the 4th week and 8.8% at 8th week. Besides, in BDS EN standard for PCC 42.5N cement it is decreased 16.8% at the 4th week and 21.1% at 8th week. For OPC 52.5N cement in BDS EN standard it is decreased 11.7% at the 4th week and 14.5% at 8th week. All the values satisfy the required standard value specified in ASTM, BDS EN. Fig. 15 shows 7 Days Compressive strength (MPa) of cement at different periods.

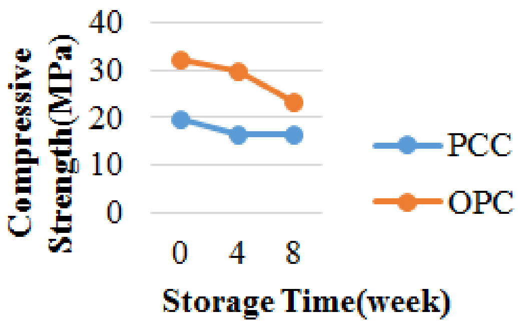

Fig. 13. 2 days compressive strength (MPa) of cement by BDS EN method at different periods.

28 Days Compressive strength: 28 days compressive strength in ASTM standard for PCC 42.5N cement is decreased 3.7% at 4th week and increased 1.4% at the 8th week. For OPC 52.5N cement in ASTM standard is decreased 8.2% at the 4th week and 2.2% at the 8th week. Again, in BDS EN standard for PCC 42.5N cement, 28 days compressive strength is decreased 10.4% at the 4th week and 12.7% at the 8th week. For OPC 52.5N cement, 28 days compressive strength is decreased 1.33% at the 4th week but increased 6.3% at the 8th week. All the values satisfy the required standard value specified in ASTM, BDS EN except the compressive strength value in BDS EN standard of PCC 42.5N cement at 8th week; which is 0.08 MPa less from the required value. Fig. 16 shows 28 Days Compressive strength (MPa) of cementat different periods.These experimental results show that Compressive strength of cement didn't vary significantly until 8th week from the production date of cement, because the quality of cement was good during that period.

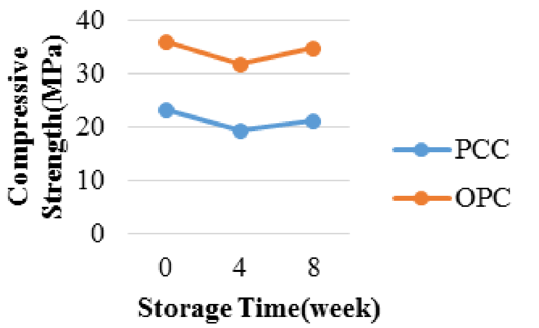

Fig 14. 3 days compressive strength (MPa) of cement by ASTM method at different periods.

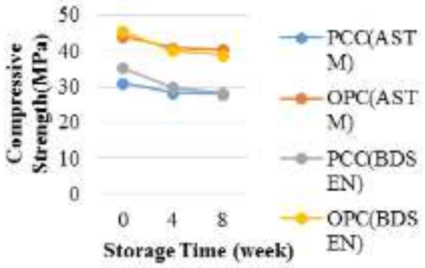

Fig 15. 7 days compressive strength (MPa) of cement at different periods.

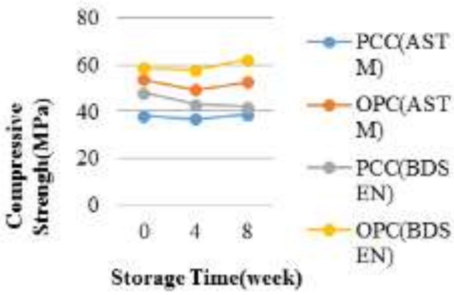

Fig. 16. 28 days compressive strength (MPa) of cement at different periods.

IV. Conclusion And Recommendation

From the observation of physical properties of same type of cement sample with the course of time, it is found that there is no remarkable degradation of cement sample during this period. The variation of the values of 28 days compressive strength of different months is not very large and this variation is occurred due to experimental error. From the test results, it can also be said that the cement sample remains perfect for use till 8th week from the manufactured date of the cement sample. So it is recommended that after 8th week from the manufacturing date of cement, the physical properties need to be tested to find out the quality of the cement. Otherwise, degradation of quality of cement may lead to an adverse impact on construction industry.

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