Impacts of Type 1L Cement on Properties of Cement Pastes for Use in Well Applications

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Table of Contents

Executive Summary

This report presents a study of the use of Type 1 and Type 1L cements for use in wellbore applications in New Jersey. A broad range of testing was done to examine the performance of the systems, including unconfined compressive strength, shrinkage, setting time, flowability, electrical conductivity, and permeability. Testing was completed following applicable standardized methods and quality assurance procedures. This work was completed at the request of the NJ Department of Environmental Protection (NJDEP) due to the imminent switchover, industry wide, from Type 1 cement to Type 1L; which will result in low availability of Type 1 cement. N.J.A.C. 7:9D “Requirements for Well Construction and Maintenance; Sealing of Abandoned Wells” currently limits the type of portland cement that can be used to traditional portland cements (i.e, Types 1, II, III and V), but does not allow the use of Type 1L. Therefore, the testing presented in this report is necessary to determine if Type 1L can be used in place of Type 1.

Results of this study indicate that the Type 1L systems perform similarly to the Type 1 counterparts. Though there were significant differences in the comparative results between systems with and without bentonite, the use of the Type 1L cement in either system only resulted in minor property changes. All four systems studied performed adequately, and would meet the current performance requirements set forth within N.J.A.C 7:9D. In regard to permeability, specifically, all systems achieved a permeability lower than the requirements set forth in N.J.A.C. 7:9D by seven days after mixing of the cement with water. The bentonite systems had higher permeabilities than the non-bentonite systems, but all were several orders of magnitude lower than the performance requirements. The use of Type 1L cement resulted in lower permeability at day 7 after casting compared to the Type 1 systems. These results indicate that the Type 1L cement should be viable for use by the well drilling industry in wellbore sealing activities.

A field trial using Type 1L cement was also completed and results are presented in this report. The specimens collected during the field trial performed adequately, and met NJDEP requirements, despite performing worse than the laboratory-prepared samples. Based on observations made during the field trial, it is unlikely that the correct water to cement ratio (0.46) was used in the field, resulting in lower strength and higher permeability. However, it is confirmed that the permeability of Type 1L still met the requirements of N.J.A.C. 7:9D.

Introduction

Background Information

Hydraulic cement is a common material used to seal the annulus between the wellbore casing and the surrounding rock or soil, thus providing long-term wellbore integrity. Cement provides bonding strength when saturated and results in low permeability (hydraulic conductivity) [1]. This fundamental property plays a key role in preventing leakage of hazardous materials and fluid movement between the delamination of the cement-annulus-casing in wells and underground geo-engineering systems. Additionally, grout systems can be used to plug wellbores and permanently seal underground infrastructures (such as oil or gas reservoirs) that are no longer in operation to prevent vertical migration of contaminants. Wellbore cement must have several important properties to ensure wellbore integrity. Some of these properties include strength, permeability, hydraulic conductivity, porosity, setting time, stiffening, flowability, density, chemical resistance, adhesion, and expansion.

There are certain types of neat wellbore cement specified and approved for the final application in wells by the NJDEP according to section N.J.A.C. 7:9D [2]. In particular, N.J.A.C. 7:9D Appendix A, has specified the acceptable neat portland cement mixture designs for systems without and with sodium based bentonite; these are reproduced in **Table 1** and **Table 2**, respectively. Following the specified mixture designs presented in the aforementioned tables, all grout materials must also have a permeability of 1 x 10^{-7} cm/s or less, as measured according to the process described in ASTM D5084 [2], [3].

Table 1. Allowable neat portland cement[1] mixture design per NJ specifications for non-thermally enhanced grouts

Type of cement	Cement (lbs)	Water(gal)	Target density (lbs/gal)	Acceptable density/range (lbs/gal)	Water/cement ratio
I and II	94	5.2	15.6	15.0 to 16.3	0.46
III	94	6.3	14.8	14.2 to 15.5	0.56

[1] Approved for use in saltwater environments

Table 2. Allowable neat portland cement[1] with sodium-based bentonite mixtures per NJ specifications

Percent bentonite	Bentonite (lbs)	Cement (lbs)	Water (gal)	Target density (lbs/gal)	Acceptable density/range (lbs/gal)	Water/cement ratio
5.3	5	94	8.3	13.9	13.4 to 14.5	0.74

[1] Aproved for use in saltwater environments

Type 1L cement, commonly referred to as portland limestone cement (PLC), is a specialized type of pre-blended hydraulic cement that incorporates limestone in addition to the traditional ingredients of ordinary portland cement (OPC). Limestone, a natural sedimentary rock rich in $CaCO_3$, is finely ground and blended with clinker, gypsum, and other additives during the cement manufacturing process. Type 1L cement contains a lower clinker content of ~90% compared to the OPC and limestone content is ~10%, conforming to the standard ASTM C595 [4], resulting in reduced embodied CO_2 emissions.

One of the most important reasons why Type 1L is used is it has limestone content which is a popular environment-friendly filler material, allowing the reduction in clinker content without significantly impacting properties. The systematic and balanced use of limestone can effectively reduce the carbon emission rate during manufacturing, transporting, and operation processes, resulting in decreasing global warming potential (GWP) [5], [6]. Another reason to use Type 1L cement is that limestone has several physical advantages such as filler effect, dilution effect, and improved nucleation of the main hydration product C-S-H [7], [8], [9]. As a result, the initial porosity of the mix is improved due to its filler effect, and the water demand is reduced maintaining a persistent workability during the well application.

The use of Type 1L instead of Type 1 as a cement paste has various implications on the physical, chemical, and mechanical properties. Due to its smaller particle size distribution, Type 1L requires less water for the desired consistency than the relative Type 1 cement. Previous research [10] reported that there is an average decline of required water content by 0.5% with the addition of 5% limestone in the mixture. Furthermore, PLC having ~10% limestone reduces the average water demand by up to 25% [11]. The presence of limestone in Type 1L has been reported to decrease the plastic viscosity and increase the flowability and improve the standard consistency [12]. Type 1L cement has also been found to increase the compressive strength with the same w/c ratio as the Type 1 system [11], however, it can impact the compressive strength more adversely with increasing w/c ratio. In concrete systems, the presence of limestone has also been reported to decrease the electrical resistivity within a range of 5-15% [11], however, it is not widely practiced for cement paste systems. Recently, the availability of Type 1 cement has become scarce within the United States as cement plants switch from making Type 1 cements to making only Type 1L cements, for environmental reasons. Therefore, it is of utmost importance that agencies that rely on Type 1 cements ensure that Type 1L will meet their standard performance protocols.

Bentonite is a natural clay that is made up of montmorillonite minerals [13], [14], [15]. It has a unique chemical structure that enables it to absorb water and swell beyond its original volume. The swelling capacity of bentonite is highly dependent on the salinity of the water, compaction of the bentonite, temperature, and physical boundary conditions. However, free expansion of sodium-based bentonite has been shown an increase in volume of up to 10 times its original volume within a few hours after being mixed with water. The swelling pressure and strain of bentonite are effective for underground seals due to their absorbent and buffer properties [16], [17]. However, one limitation of bentonite application is its failure at temperatures beyond 100°C [18]. In cementing applications, bentonite is commonly used in cement grout as an underground seal for geo-engineering and civil engineering such as wellbore plugging, and drilling fluids, and as a natural sealant for repositories, ponds, and landfills. It can enhance the viscosity and the thixotropic properties of cement grout during placement which ensures proper zonal isolation and prevents fluid migration in underground infrastructure. When bentonite is mixed with cement, the bentonite absorbs water and expands in the fresh paste, making the paste denser and potentially reducing its capillary pores. During cement hydration, the surface of bentonite grains acts as nucleation sites and promotes the formation of hydration products which prevents external water from entering the capillary pores due to swelling. The hydration products increased the tortuosity of water movement paths and refined paste pores, hindering water movement [19]. Additionally, the pozzolanic reaction between bentonite and calcium hydroxide generates more calcium silicate hydrate (C-S-H), the main strength and durability compound in hydrated cement-based materials, densifying the matrix and reducing the permeability of the paste [19].

In terms of the widespread application of Type 1L cement, many industries such as road and building infrastructures are currently using it for its reduced carbon potential and improved strength development compared to normal Type 1 cement. However, very little work has been done studying Type 1L and Type 1L-bentonite system performance for underground structures such as wellbores. Some research works were previously carried out using limestone and bentonite as additives with Type 1 cement for wellbore application [12], [20]. One of the key areas of interest is the long-term durability of the Type 1L in the wellbore, particularly under high in-situ stresses and temperature conditions. Another area that requires more research is the impact of Type 1L on wellbore stability in freeze-thaw and saline environments and how it will interact with other fluids (oil spillage, acidic liquids from septic systems, etc.) Additionally, more research is needed to determine the optimal concentration of Type 1L for different types of drilling operations and formations.

Project Design, Methods, and Quality Assurance

Materials

Cement

Type 1 ordinary portland cement (OPC), which conformed to the ASTM C150 specification [21], was used in the presented work. Additionally, a blended hydraulic PLC, Type 1L, was used following the standard ASTM C595 [4]. The limestone content of the Type 1L cement used for this work was 9.16%. As provided by the manufacturers, quantitative chemical oxide analyses of Type 1 and Type 1L cement are presented in **Table 3**.

Sodium-Based Bentonite

As an extender, sodium-based bentonite was used in the mix design for the preparation of Type 1-bentonite and Type 1L-bentonite. Bentonite, derived from the smectite group of clays, is renowned for its remarkable swelling properties [22]. Its primary constituent, montmorillonite, possesses a double diffusion layer, contributing to its distinctive swelling behavior [22]. In this study, the size of bentonite clay particles achieved from the dry screen analysis from the supplier was 0.0937 in. (2.38 mm) or 8 mesh. The use of bentonite particles was made to analyze different physical properties of two different paste systems and there was no partial replacement of cement.

TABLE 3. Quantitative oxide analysis of Type 1 and Type 1L cements

Constituent	Amount (%)	
	Type 1	Type 1L
SiO_2	18.9	17.67
Al_2O_3	4.72	4.36
Fe_2O_3	2.29	2.11
CaO	62.84	61.74
MgO	3.08	2.9
SO_3	3.45	4.01
Na_2O	0.3	0.37
K_2O	1	0.92
Limestone	3.7	9.16
Loss on Ignition	2.73	5.29
Insoluble Residue	0.16	0.18
Content of CaCO3 in Limestone	95.8	95.4

Experimental Methods

Mixture Design

In the comprehensive study, four (04) different cement paste systems (CPS) were used to prepare different CPS to evaluate and compare different physical and mechanical properties. A target density of 1870 kg/m^3 was maintained for Type 1 and Type 1L, while it was kept at 1665 kg/m^3 for Type 1-bentonite and Type 1L-bentonite. The systematic selection of different CPS was based on different types of cement and w/c ratios (see **Table 4**).

TABLE 4. Composition of different cement paste systems

Cement paste systems	water/cement ratio	Additional plugging agent	Target density (kg/m3)
Type 1	0.46	N/A	1870
Type 1L	0.46	N/A	1870
Type 1- bentonite	0.74	Sodium-based bentonite	1665
Type 1L- bentonite	0.74	Sodium-based bentonite	1665

In addition, a special plugging and sealing agent, sodium-based bentonite, was introduced in Type 1-bentonite and Type 1L-bentonite cement systems for a higher water-to-cement (w/c) ratio of 0.74. The quantity of bentonite was taken as 5.3% of cement in the respective mixtures.

Sample Preparation and Mixing Procedure

As outlined in **Table 4**, the mixing of materials for each CPS was carried out following the standard ASTM C305 [23]. Type 1 and Type 1L cement were mixed with water separately, maintaining a w/c ratio of 0.46. After the materials were placed in an electrically operated mechanical mixer, a mixing operation was carried out at a speed of 140±5 rpm for 30 seconds and 285±10 rpm for 60 seconds following a pause of 15 seconds. For Type 1-bentonite and Type 1L-bentonite, sodium-based bentonite was taken at 5.3% of the respective cement by mass. Weighed bentonite was then mixed with cement and water following a w/c ratio of 0.74. The mixing operation for Type 1-bentonite and Type 1L-bentonite was like that of Type 1 and Type 1L.

Consistency and Viscosity

After the mixing operation, the consistency and viscosity of different CPS were evaluated, and results were recorded. Through setting time tests using Vicat apparatus conforming to ASTM C191 [24], the consistency of all samples was measured to learn more about the workability and setting time of the produced cement paste. Given the viscosity of freshly produced CPS, a graduated Marsh funnel device was employed to determine the viscosity of different slurries. 1.5 L slurry samples from each CPS were taken and the required flow duration was recorded to fill a volumetric space of 1 L conforming to ASTM D6910 [25].

Flowability

Following a similar mixing operation as governed by ASTM C305 [23], the flowability of all CPS samples was evaluated as per the standard ASTM C1437 [26]. A rigid flow table having a diameter of 10 in. was used following ASTM C230 [27]. Freshly prepared CPSs were placed and tamped accordingly in a conical mold having a height of 2 in. (50 mm), a top and a bottom diameter of 2.75 in. and 4 in. (69.85 mm and 100 mm), respectively. The mean flow value was recorded by dropping the flow table at a rate of 25 times in 15 seconds. However, modifications were made to Type 1-bentonite and Type 1L-bentonite since cement pastes were overflowing from the table due to high water content in both systems before the drop was initiated. For these systems, the procedure was repeated at 30-minute intervals to track changes in the rate of flow over time after the mixing operation.

Early Stiffening Test

Following the standard ASTM C451 [28], the rate of early stiffening for different paste systems was measured. Initially, 500 gm of cement (Type 1 and Type 1L) was taken for each trial during the determination of water content to produce a hydraulic cement paste having an initial penetration ranging from 1.10 in. to 1.42 in. (28 mm to 36 mm). For bentonite-based paste systems, bentonite was taken 26.5 gm (5.3% of cement mass) and mixed with cement before mixing water. Cement and water were then mixed at a speed of 140±5 rpm for 30 seconds in a dry bowl of a mechanical mixer. After the initial mixing for 30 seconds, unmixed materials were scraped and remixed with the paste from the side of the bowl within 15 seconds. The paste was again mixed for 150 seconds at a speed of 285±10 rpm. Upon finishing the final mixing, the cement paste was immediately shaped into a ball and filled in a conical ring in the Vicat apparatus. Then the initial penetration was measured 20 seconds after mixing the paste by releasing the plunger of 0.39 in. (10 mm) diameter on the paste surface. The plunger was allowed to penetrate through the paste 30 seconds before the initial penetration was measured. Following the same procedure, the final penetration depth was measured after 5 minutes± 10 seconds after the initial mixing.

Compression Test

For the uniaxial compressive strength test, prepared CPSs were batched using 2 in. (50 mm) cubic specimens following the mixing operations as per ASTM C109 [29]. Throughout the batching operation and materials handling, the room temperature was recorded and kept at 23±3°C. Following the batching of cubic specimens, all samples were immediately wrapped in wet hessian cloth and taken to be cured for 24 hours in the moist cabinet according to the standard ASTM C511 [30]. Following a 24-hour curing period, all CPS samples were removed from the mold and placed into the hydrated-lime saturated water storage tank to be further cured at 22-23°C for different curing periods. Throughout the procedure, three (3) samples from each CPS were prepared for each specified curing periods, such as 3, 7, 14, 28, 56, and 90 days.

Surface Resistivity

To measure the electrical conductance through different CPSs, a surface resistivity (SR) test was carried out on each sample conforming to AASTHO T358 [31]. **Figure 1** depicts a schematic of the Wenner probe and specimen setup in the laboratory. Initially, the mixing procedure was followed according to ASTM C305 [23]. Three cylinders from each CPS were prepared and batched following the procedures as mentioned in ASTM C39 [32] and the size of the cylinder was 4 in. by 8 in. (100 mm by 200 mm) After 24 hours of curing in the moist cabinet, all samples were demolded from the cylinders and four straight lines labeling 0°, 90°, 180°, and 270° were drawn on the circumference having a centerline.

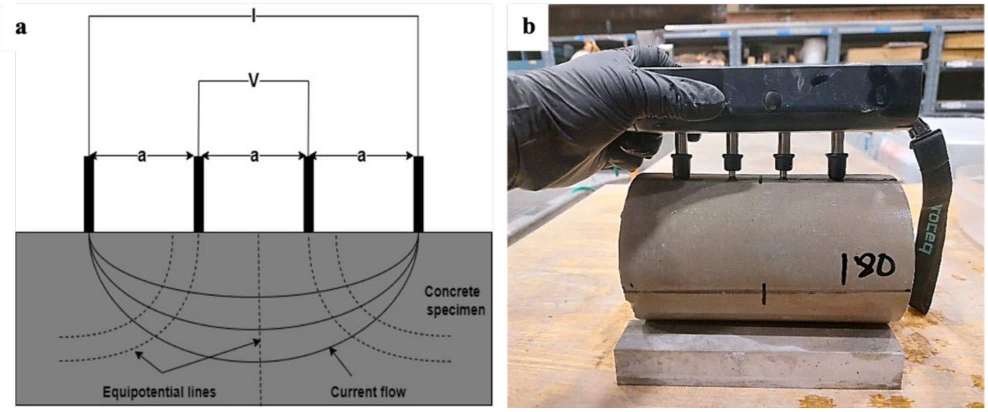

FIGURE 1. (a) Schematic of 4-point Wenner probe, (b) resistivity measurement of concrete cylinder specimen

Marked samples were then allowed to be wet-cured in the water storage tanks at 22-23°C [33]. As the prescribed curing periods reached consecutively 7, 14, 28, 56, and 91 days, the surface of the specimens was brought to a saturated surface dry (SSD) condition immediately before the test by cleaning the exterior surface with a wet towel. According to AASTHO T358 [31], a four-point Wenner probe was carefully placed longitudinally on the labeled circumference making an equidistant position of two pins of the inner probe from the marked centerline. Initially, a Wenner probe started measuring the 0°-labelled position, and subsequent readings were recorded by rotating the cylinder counterclockwise from 0° to 90° and so on. Repeating the steps from 0° to 270° labeled position, two readings were taken from each labeled position to get an average SR value of three reference cylinders. All measurements were recorded within an average time of 6 to 7 minutes to minimize variability in results and to complete the entire test before the cylinder turned to dry state from SSD condition.

Rapid Chloride Penetration Test

Furthermore, cylindrical specimens were prepared to measure the rapid chloride penetration test (RCPT) of hardened CPSs to evaluate a rapid manifestation of the permeability of chloride ions according to ASTM C1202 [34]. One cylindrical replicate from each CPS was prepared and batched in a 4 in. by 8 in. (100 mm by 200 mm) cylindrical mold following the procedures mentioned in ASTM C305 [23] and ASTM C39 [32]. After the curing period of 28 days, specimens were taken out of the storage tank and three samples were cut from each specimen using a saw cutter. The height and diameter of each specimen was 2 in. (50 mm) and 4 in. (100 mm), respectively. Following a 1-hour drying period, rapid-setting epoxy coating was applied on the circumference of each saw-cut cylindrical core and allowed to set properly on the surface. To ensure consistency, the specimens were vacuum-saturated before testing, adhering to the guidelines outlined in ASTM C1202 [34]. After an 18-hour soaking period, cylindrical cores were positioned between two cells each filled with a specific solution: one with a 0.3 N NaOH solution and the other with a 3% NaCl solution. Two cells were connected to a 60-volt power supply in the RCPT measuring device.

Throughout a duration of 6 hours, the flow of current through the specimens was monitored and documented.

Permeability (Hydraulic Conductivity) Test

In this study, four different types of cylindrical system types were prepared to represent the various compositions of cement-based systems (Type 1, Type 1L, Type 1-bentonite, and Type 1L-bentonite). The dimensions of the cylindrical plastic mold were 3 in. (75 mm) diameter by 6 in. (150 mm) height. An automated LoadTrac Triaxial Testing System was used to test the permeability of the hardened cement paste (system types). The permeability testing system uses flexible-wall permeameters, as specified in ASTM D5084-16a [3]. The flexible wall permeameter is used to test system types with permeability equal to or less than x10^{-6} m/s under a unit gradient of 0.0864 m/day. Notably, for the effective use of a flexible-wall permeameter, the system types must undergo full saturation before compaction as suggested in ASTM D5084-16a [3]. A confining pressure of 4.35 psi was applied to the cell, and additionally, 2.90 psi influent and effluent pressures were applied to the systems to allow permeant water to flush through the flow system. After ensuring that all visible air was removed from the flow lines by jogging the pressure systems, the control valves were closed, and the test was set to run autonomously. A cell pressure of 44.8 psi and a backpressure of 39.9 psi were applied during the consolidation phase to consolidate each system type. Subsequently, a cell pressure of 44.8 psi, an influent pressure of 40.9 psi, and an effluent pressure of 39,9 psi were used to permeate the system types. The representation of the permeameter and the experimental setup are presented in **Figure 2**.

When the test was completed for each system type, the data was imported into Microsoft Excel. The permeability (cm/s) was then plotted against time (s), and the steady-state method was used to accurately measure the average permeability of the system types, as represented by the red bars in **Figure 3**.

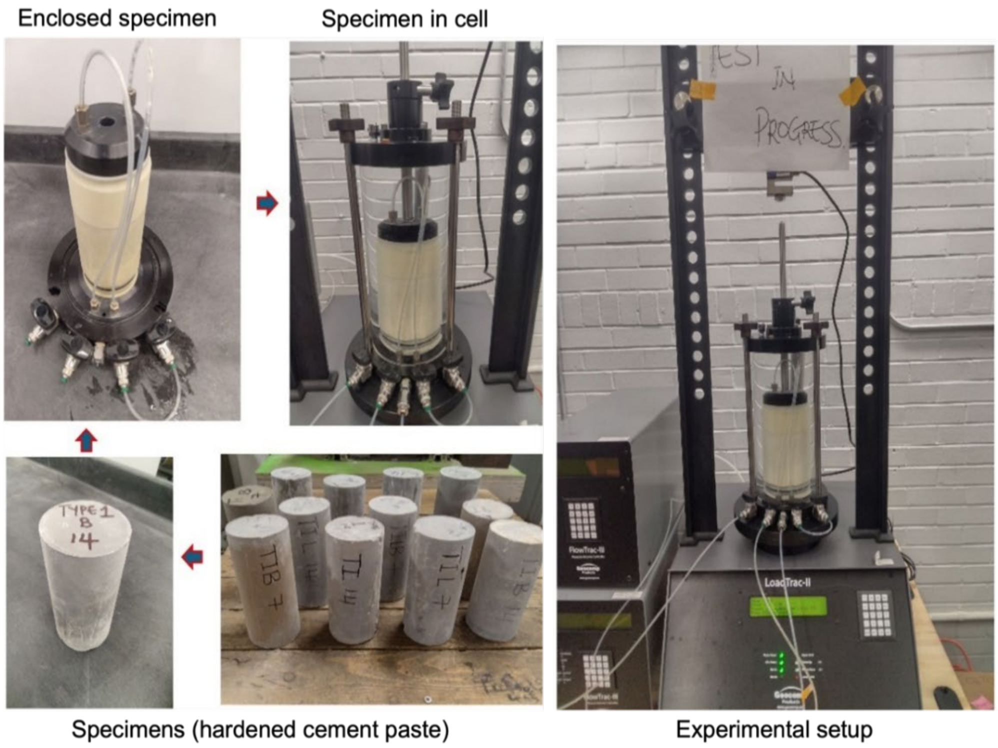

FIGURE 2. Experimental set up of the permeameter

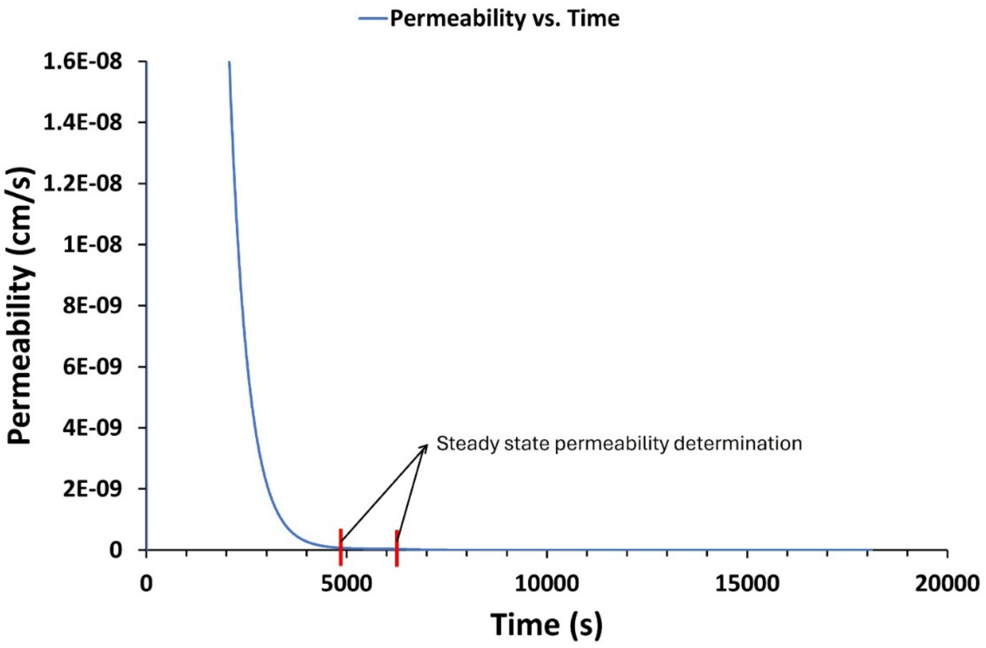

FIGURE 3. Determination of permeability of cement-based systems

Drying Shrinkage

Drying shrinkage measures the length change of an unrestrained paste specimen that has been allowed to dry. During drying, water leaves the pores in the paste, resulting in the collapse of the pore space and macroscopic volume change. In a system under restraint (which includes most concrete placed in the field), the shrinkage results in a buildup of internal stresses. These stresses can result in cracking if the tensile strength of the cement paste has not exceeded the internal stress [35]. For this work, ASTM C157 was followed to measure the free shrinkage of cement paste systems [36].

Results and Discussion

Mechanical Properties

Compressive strength

The compressive strength of the four different CPS has been measured for the curing times of 3, 7, 14, 28, 56, and 90 days. According to the results obtained from the system types without bentonite (Type1 and Type 1L), it was found that at 3 days, the average compressive strength of Type1 and Type 1L system types were 5360 and 5390 psi, respectively. At 7 days, the compressive strength of Type1 and Type 1L were 6050 and 5720 psi, respectively; and at 90 days, the compressive strength of Type1 and Type 1L system types were 9480 and 9390 psi, respectively. It is evident from **Figure 4** that, in most cases, Type 1L system offered higher strength on 3 days, 14 days, 28 days, and 56 days, respectively, compared to Type1. The trend is supported by previous research [37] in the case of Type1 and Type 1L containing up to 10% limestone content. In this study, as evidenced by the mill report from the manufacturer, the presence of 9.16% of limestone in Type 1L cement may have influenced the accelerated growth of nucleation sites for the C_3S phase led to both early stage and long-term strength development [38], [39]. However, there was still a gradual increase observed for both Type 1 and Type 1L from after 28 days through 90 days. One possible reason for the increase in compressive strength could be attributed to the separate batching and preparation of the two systems. The initial four batch replicates (ranging from 3 days to 28 days) were cast on a single day, while the subsequent replicates were batched and prepared on a different day. The deviation may result from differential laboratory temperatures, curing conditions, moisture content, etc. [40], [41]. These differences are normal in concrete laboratories.

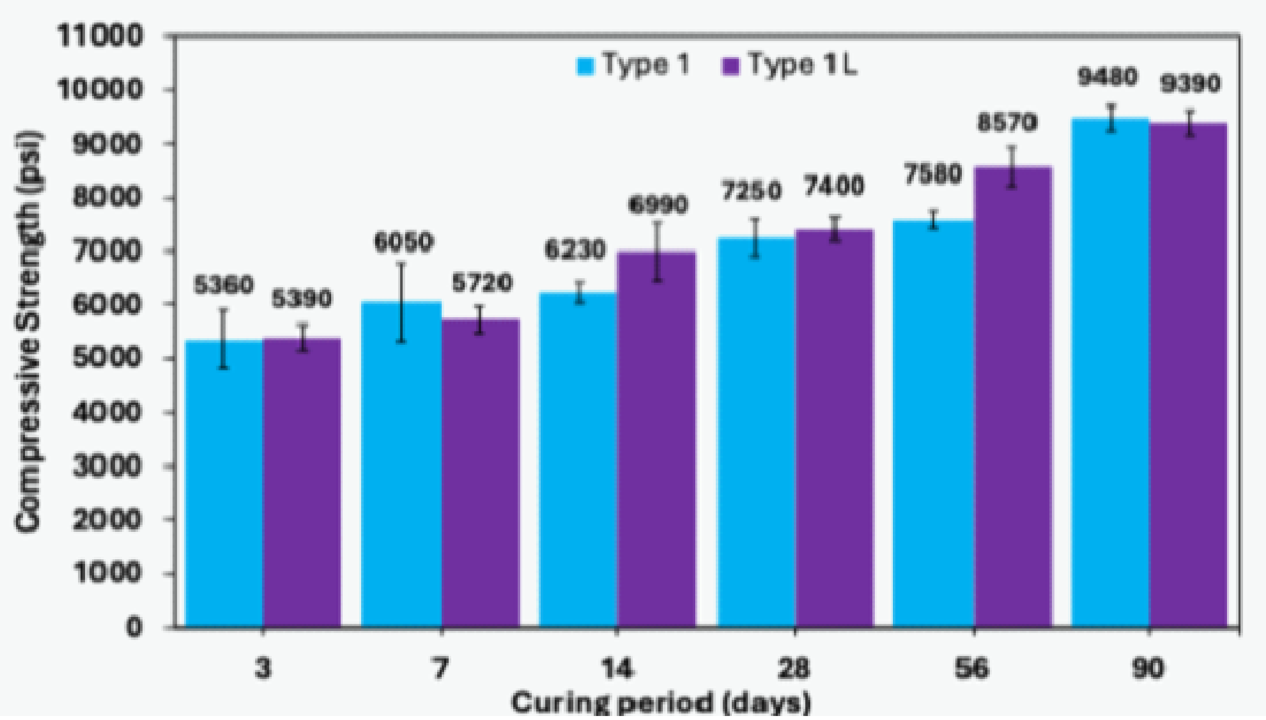

FIGURE 4. Compressive strength of cement-based systems (Type 1 and Type 1L)

The development of strength for Type 1-bentonite and Type 1L-bentonite in **Figure 5** was somewhat opposite compared to the other two systems mentioned above. It was found that at 3 days, the compressive strength of Type 1-bentonite and Type 1L-bentonite system types were 2120 and 1660 psi, respectively. At 7 days, the compressive strength of Type 1-bentonite and Type 1L-bentonite were 2820 and 2330 psi, respectively; and at 90 days, the compressive strength of Type 1-bentonite and Type 1L-bentonite system types were 3920 and 3420 psi, respectively.

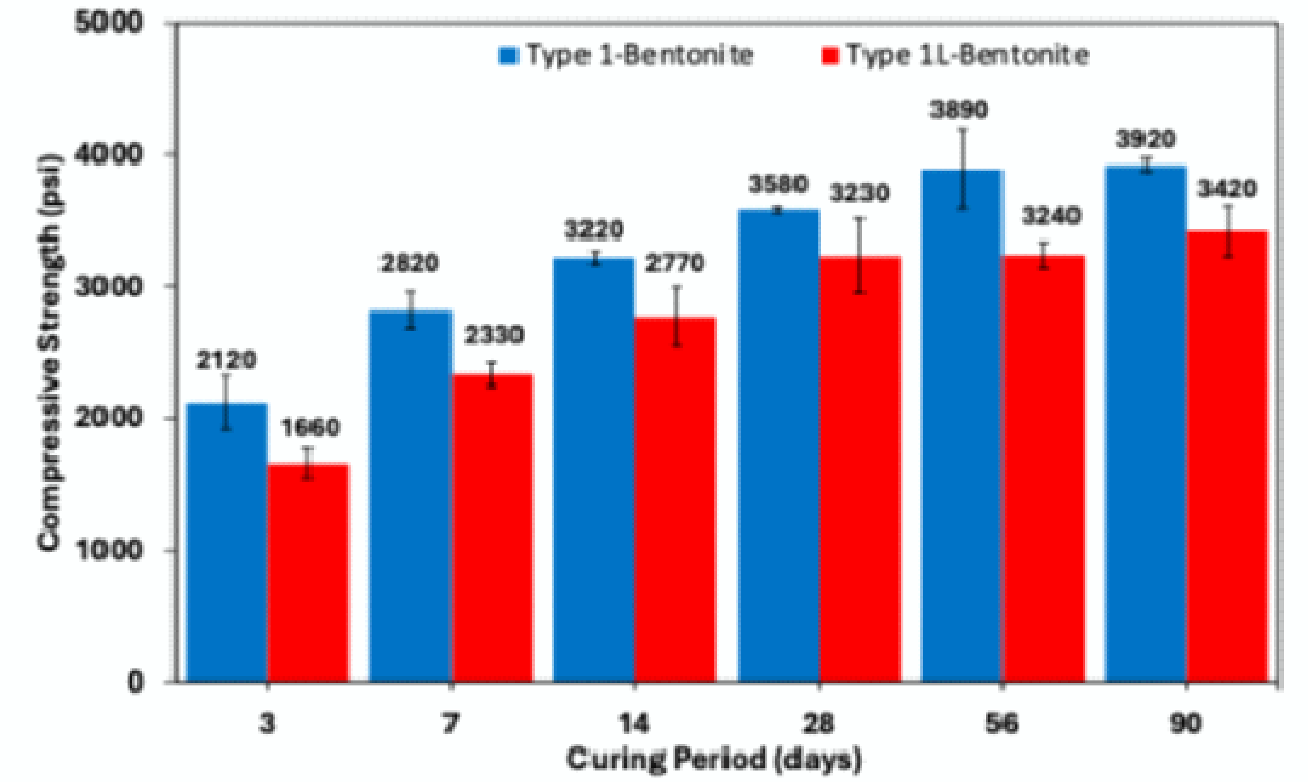

FIGURE 5. Compressive strength of cement-based systems (Type 1-bentonite and Type 1L-bentonite)

The strength was significantly reduced compared to the non-bentonite systems due to the higher w/c ratio since higher water content increases the porosity of hardened cement paste [42]. In addition to the high w/c ratio, the inclusion of bentonite clay significantly reduces the compressive strength and this observation is supported by previous research [43]. This is because bentonite has a retardation effect on the hydration reactions of cement paste due to its lower pozzolanic activity with high water content. However, when comparing the difference between both bentonite systems, the Type 1L bentonite system's average compressive strength was slightly less than that of the Type 1 bentonite system.

Fresh Properties

Setting Time Test

The workability of fresh CPS via setting time test was evaluated and results are presented in **Table 5**. Differences in the values of initial and final setting time are observed from the presented data. Initial set time indicates when the flowability of a cement paste system becomes unworkable, and final set time indicates when the cement paste system becomes rigid enough to start bearing load without deforming.

Table 5. Setting time test results by Vicat needle

System type	Water/Cement Ratio	Bentonite (%) by mass of cement	Initial Setting Time (minutes)	Final Setting Time (minutes)
Type 1	0.46	N/A	320	510
Type 1-Bentonite	0.74	5.3	473	765
Type 1L	0.46	N/A	369	565
Type 1L-Bentonite	0.74	5.3	518	810

In Type 1, Type1 portland cement was employed, and the observed initial and final setting times were recorded at 320 minutes and 510 minutes consecutively. As the type of cement was changed keeping the same w/c ratio, for instance, Type 1L PLC in Type 1L, an extension for both setting times was recorded.

In addition, cement paste took a longer period to lose its plastic state for Type 1-bentonite and Type 1L-bentonite when 5.3% of bentonite by mass of cement was introduced, compared to Type 1 and Type 1L. The combined effect of cement-bentonite and high-water content also delayed the time required to start achieving the strength at the solid state of the paste. Compared to Type 1-bentonite, both initial and final setting times were delayed by 45 minutes when the mixture contained PLC and bentonite in Type 1L-bentonite.

Flowability and Viscosity

Using the CPS presented in **Table 4**, a flow table test was conducted to study the impact on the overall fluidity for varying w/c ratio and the addition of bentonite with OPC and PLC system. According to the presented flowability data in **Table 6**, the fluidity of different CPS varies with the type of cement, water content, and the presence of bentonite in the system.

Table 6. Flowability of different cement paste systems

System type	Flowability (%)	Time required toreach measurable flowability (minutes)	Viscosity (sec)
Type 1	129	< 5	NA**
Type 1-Bentonite	140*	270	81
Type 1L	136	< 5	NA**
Type 1L-Bentonite	141*	300	41

* Longer time required to achieve the desired flowability may be due to high water content

** Paste did not flow through the orfice of Marsh Funnel, may be due to denser consistency, led to failure in measuring the viscosity

The paste becomes more flowable with the addition of Type 1L cement having a prescribed limestone content of 10% as per ASTM C595 [4]. The flowability was higher for Type 1L than that of Type 1, and previous studies also revealed that blended limestone content increases the fluidity compared to OPC cement [12], [20]. However, the difference in flowability between the two mixtures was within standard variability of the test, and so the difference is not significant, indicating that similar flowability can be expected when using Type 1L cements in the mixture designs mandated for well sealing in New Jersey. The high-water content in the bentonite systems led to an overflow for both CPS containing bentonite, and that led to the modification of the regular test to a measurable result, as noted in 3.2.4.

Change of flow rate over time was monitored every 30 minutes and the time the Type 1-bentonite did not overflow to the table was after 270 minutes of initial cement-water contact. However, Type 1L-bentonite required 30 additional minutes to reach a measurable flow compared to the Type 1-bentonite CPS. This outcome aligns with the observation made in [44] where the actual water demand is less for PLC than OPC. However, it is unclear how significant this difference is, as the modified test method has no precision and bias statement, and the research team did not perform an adequate number of replicates to study statistical significance. Practically, however, this indicates that users in the field should expect the fluidity of systems made with Type 1L to last longer.

Beyond the flow test, Marsh Funnel measurements were also done to examine the viscosity and flow characteristics of the systems. Due to the higher viscosity of Type 1 and Type 1L systems, it was difficult for the paste to flow through the orifice having an inside diameter of 0.1875 in. (4.76 mm) of the Marsh funnel tube. As a result, measurements were not made for the Type 1 and Type 1L CPS. Type 1-bentonite was denser in its fluid concentration compared to Type 1L-bentonite and it led to an increased viscosity of 81 seconds for 1 L paste to flow compared to Type 1L-bentonite. Since the particle size distribution of limestone in Type 1L-bentonite is smaller compared to the clinker size of Type

1-bentonite, increased viscosity was observed for Type 1-bentonite having a Type1 ordinary cement. This result regarding the viscosity matched the output presented in previous research [45]. As a result, the Type 1L-bentonite system offers great flowable properties with reduced viscosity compared to the other three paste systems.

Early Stiffening of Hydraulic Cement

For hydraulic cement paste to be workable at the site after the mixing operation, it is necessary to determine the extent to which a freshly produced cement paste develops a certain amount of stiffness at the early stage. **Table 7** represents different paste systems, the quantity of bentonite, and the required water content (w/c ratio) to study the early stiffening rate conforming to the ASTM C451 [28]. Type 1L requires less water (w/c=0.29) to produce an initial threshold consistency of 1.26 in. ± 0.08 in. (32 mm ± 2 mm) when compared to the Type 1 system (0.32). The final penetration after 5 minutes±10 seconds was 68.6% for Type 1 while it was 57.6% for Type 1L. In addition, after the bentonite was mixed, the final penetration was recorded at 84.8% for Type 1-bentonite (w/c=0.36) while it was 44.4% for Type 1L-bentonite (w/c=0.32).

TABLE 7. Rate of early stiffening of cement paste systems

System type	Cement (gm)	Bentonite as 5.3% of cement (gm)	Water/cement ratio	Final penetration (%)
Type 1	500	N/A	0.32	68.6
Type 1-Bentonite	500	26.5	0.36	84.8
Type 1L	500	N/A	0.29	57.6
Type 1L-Bentonite	500	26.5	0.32	44.4

Based on the above result, the rate of early stiffening is higher for both Type 1L systems (with and without bentonite) indicating a rapid formation of resistance to deformation compared to both Type 1 systems.

Durability Properties

Surface Resistivity

A 4-point Wenner probe was used to study the development of surface resistivity (SR) which is a representative value indicating permeability in cement-based systems – with higher resistivity indicating lower permeability. The graphical representation in **Figure 6** indicates that both Type 1 and Type 1L gradually developed their resistance although their respective values over time were within the expected variation for the test method. For instance, the resistivity was reported to be 0.51 kohm-in for both Type 1 and Type 1L systems at 7 days. However, Type 1 exhibited more resistivity, compared to Type 1L at later curing periods, but a difference of 0.12 kohm-in is relatively small compared to the expected variation in the test method.

Given the above result, Type 1L provides comparatively higher permeability compared to Type 1 cement, although the difference is minor. Since Type 1L is a PLC having a limestone content of 10%, it is widely considered to be responsible for creating a more porous paste matrix given the provided water content when compared to Type 1 resulting in a decreased resistivity. Moreover, limestone is an inert material and requires less water [20] compared to OPC and this led to an extended porous network despite the same w/c ratio. However, SR value represents a comparative value and does not represent a quantitative measurement of permeability.

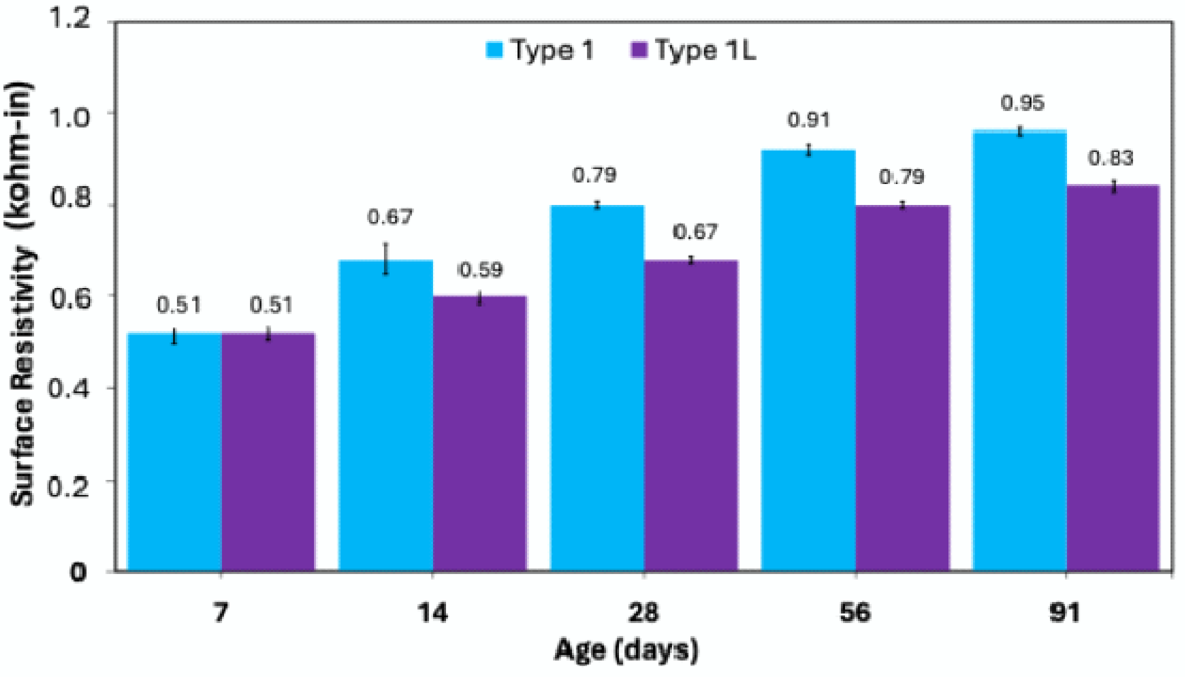

FIGURE 6. Surface resistivity of cement-paste systems (Type 1 and Type 1L)

Table 8 provides a correlation between SR values and chloride ion permeability. In addition, the resistivity of both Type 1-bentonite and Type 1L-bentonite was found to be less than 1 kohm-cm by a 4-point Wenner probe, and therefore, no SR values were recorded. These two systems contained a higher w/c ratio of 0.74 and bentonite clay that made the matrix of hardened paste extremely porous, as a result, the resistivity was extremely low for both cases, likely because of the amount of water available for charge to pass through the system. By experimental definition, the result of the SR analysis supported the finding reported in [46] where the electrical resistivity of hardened cement paste was below 5 ohm-m. According to **Table 8**, obtained SR values (< 4.7 kohm-in) from all systems suggest higher chloride ion permeability.

TABLE 8. Correlation between surface resistivity and chloride ion permeability

Chloride ion permeability	Surface resistivity of 4 in x 8 in specimen and prob spacing - 1.5 in (kohm-in)
	28-day
High	<4.7
Moderate	4.7-8.3
Low	8.3-14.6
Very Low	14.6-100
Negligible	>100

Rapid Chloride Penetration Test

The RCPT was unable to be completed for all systems during the testing phase. According to ASTM C1202 [34], the required time for a complete RCPT is 6 hours. However, no results were obtained since no specimens endured the 6 hours experimental period, with the system overloading before the testing period was complete. The failure might be due to the short circuit that led to the termination of current flow through cells instantaneously. For 28-day cured specimens, the test was immediately stopped for both Type 1-bentonite and Type 1L-bentonite systems due to overflow of current exceeding the tolerance limit of 500±0.2 mA [47], as specified by the manufacturer of RCPT measuring device. Meanwhile, the average passed charge was 306 coulombs for Type 1 while the test was automatically terminated within 13 minutes for exceeding the tolerance flow limit. In terms of Type 1L, the test lasted for 27 minutes while recording an average passed charge of 696 coulombs.

Like the surface resistivity test, RCPT is an electrical test that is an indicator for understanding the transport property and permeability of hardened concrete or cement paste or mortar samples. It measures the penetration resistance of such specimens against the ingress of chloride ions or any hazardous ions that might potentially affect the long-term durability. The RCPT value is highly dependent on the pore structure and moisture content of hardened cement paste and a porous system allows higher diffusion of ions through the matrix [48]. Both RCPT and surface resistivity tests represent very low values suggesting higher permeable paste systems and the observation is supported by past studies [48], [49]. However, other factors such as presence of cracks, curing temperature, and moisture condition, may have also influenced the resistance outcome. Therefore, it is somewhat still unclear why the samples passed such significant levels of charge and further investigations are necessary. Additionally, we were unable to find previous research that examined how bentonite may impact the results achieved in RCPT, and if the bentonite material may pass a charge, while still reducing permeability. Therefore, when assessing permeability, we do not recommend using RCPT for the grout systems.

Permeability of Cement-Based System

Quantitative permeability measurements were conducted on the cement-based system according to ASTM D5084 [3] for both Type 1 and Type 1L cement, with and without the addition of bentonite. The permeability results obtained are presented in **Table 9**. The systems were cured for 7, 14, and 28 days for complete hydration, and the permeability tests conducted for each system took approximately 23 hours to complete. Per these results, all systems had lower permeabilities than the maximum permeability of $1x10^{-7}$ cm/s required by N.J.A.C.7:9 [50].

TABLE 9. Permeability measurement of cement paste systems

Curing period (days)	Permeability (cm/s)			
	Type 1	Type 1-Bentonite	Type 1L	Type 1L-Bentonite
7	4.34E-11	6.28E-11	17.00E-11	8.57E-11
14	3.23E-11	1.05E-11	14.00E-11	5.08E-11
28	1.17E-11	0.29E-11	8.20E-11	1.27E-11

According to the results, the Type 1 system types cured for 7 days showed higher permeability compared to those cured for 14 and 28 days, with 28 days showing the lowest permeability. Likewise, the result obtained in Type 1L system types cured for 7 days showed higher permeability compared to those cured for 14 days and 28 days, with 28 days showing the least permeability. This would be expected as continued hydration of the cement pastes will result in a densification of the microstructure.

For system types made with bentonite (Type 1-bentonite and Type 1L-bentonite), a similar trend in permeability was noticed. The Type 1-bentonite system types cured for 7 days showed higher permeability compared to those cured for 14 days and 28 days, with 28 days showing the least permeability. Further, the Type 1L-bentonite system types cured for 7 days showed higher permeability compared to those cured for 14 days and 28 days, with 28 days showing the least permeability. It was observed that as the curing duration continued to 28 days, the permeability decreased, allowing less water to flow through the pores of the system types. Although the ability of the paste to resist fluid passage decreased with time, the rate of decrease was different for each system type.

The permeability measurement of the system types for short-term wellbore integrity is presented in **Figure 3**, and the percentage change between the early and late curing periods has been plotted to highlight the differences in permeability. In the Type 1 system types, the permeability decreased by 26%. This shows that for the Type 1 system types tested, the range of maximum and minimum permeability measurements for system types cured for 7 days and 14 days did not differ significantly, resulting in a slightly moderate percentage decrease. In the Type 1L system types, the permeability decreased by 18%.

These results show that in the Type 1L system types tested, the range of maximum and minimum permeability measurement for system type cured for 7 days and 14 days was small, resulting in the least percentage decrease. Similarly, in the Type 1L-bentonite system types, the permeability decreased by 41%. This shows that in the Type 1L-bentonite system types investigated, the system type cured for 14 days showed a significantly lower permeability compared to those cured for 7 days, resulting in a moderate percentage decrease. In the Type 1-bentonite system types, the permeability decreased by 83%. This shows that in the Type 1-bentonite system types tested, the system type cured for 14 days showed a lower permeability compared to the higher permeability for the system type cured for 7 days. The wide range between these permeability values based on the system type's curing duration resulted in a higher percentage decrease compared to the other system types. This result is consistent with previous studies [51], [52], [53], which have demonstrated that the curing period affects the microstructure and permeability of cementitious materials. The decrease in permeability for the system types containing bentonite in their matrix is an important parameter that prevents the migration of ions and fluids in wellbores and other geological repositories. This property is related to studies where bentonite and Type 1 were added to improve the pore structure of cementitious materials which reduced the permeability [54], [55].

Volume Change

Volume stability of the cement systems was measured by studying the length change of the systems in drying conditions. **Figure 7** shows the results from the tested systems when subjected to the testing parameters described in the methods section above. The results indicate that the bentonite systems experienced larger volume change compared to their non-bentonite counterparts, which is likely due to the higher water content. Drying shrinkage occurs when water leaves pores, resulting in a vacuum within the small pore that can cause the pore to collapse. Additionally, the systems with Type 1L cement have slightly higher levels of shrinkage than the systems with Type 1 cement. The main system of concern here is the Type 1L-bentonite system which has a shrinkage at 28-days over 0.4%, which is when cracking can begin to occur in cement paste systems. If the systems are properly cured during initial hydration (i.e., not exposed to the open air or allowed to dry) then shrinkage will likely not be a concern.

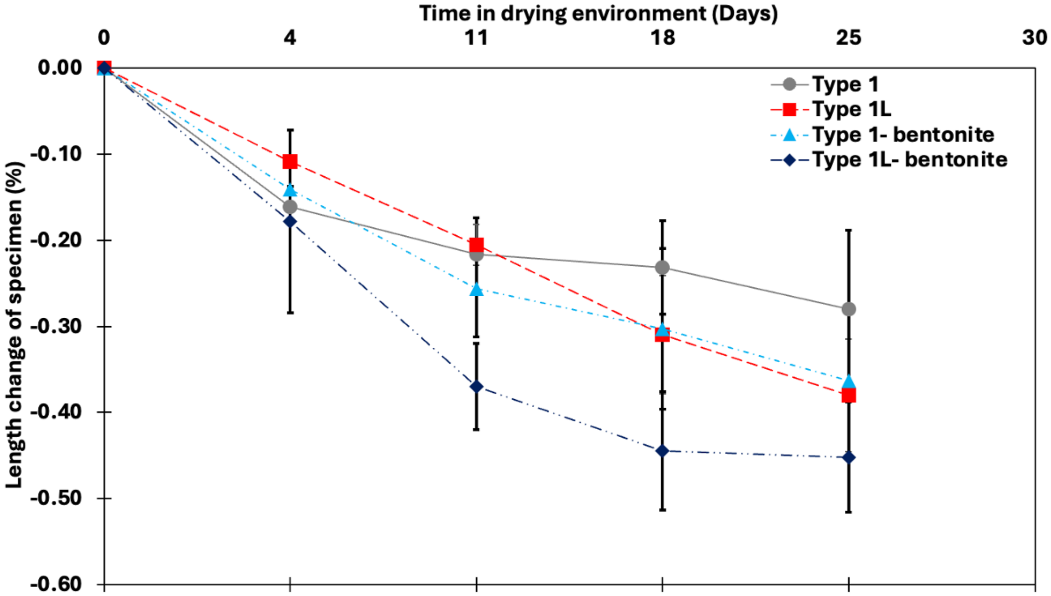

FIGURE 7. Length change due to drying shrinkage over time for all systems

Field Trials

On 15 May 2025, field trial using Type 1L cement in an actual wellbore sealing operation for the Newark Watershed located at 223 Echo Lake Road, West Milford, NJ was completed. The wellbore sealing was completed by an external contractor, not NJIT. All cement paste used in the application was batched and mixed by the contractor. NJIT sampled paste from the batched grout prior to it being placed in the wellbore. The sample of cement paste was used to create testing specimens for mechanical properties and permeability measurements were made. This section provides information on the materials used at the field trial, observations on the operations at the field trial, and results of the specimen testing. All testing and measurements followed the procedures outlined in Section 3.

Materials

The grout used for sealing Newark Water Shed well in West Milford, NJ, was produced using Athenia brand Type 1L portland limestone cement. It should be noted that the Type 1L used by the contractor was from a different manufacturer than what was used in the laboratory testing, so the chemistry and possibly the limestone content were likely different. NJIT was unable to obtain a chemical analysis of the Athenia Type 1L cement; however, the difference should have minimal impact, as both met the specification requirements set forth by ASTM C595.

The grout was mixed to have a w/c of 0.46, however as discussed in the following section, it is unlikely that this was the actual w/c. The in-situ fresh density was measured approximately 1706 kg/m^3.

Observations during In-situ Casting

During our field observations, we identified significant differences in casting and sampling procedures between on-site practices and laboratory protocols. Notably, field temperatures averaged approximately 18 °C, compared to around 23 °C in the controlled laboratory environment, potentially influencing material behavior and setting times. In the field, the preparation of the cement paste grout was conducted directly in the mixer, where cement was randomly introduced into water during mixing. This contrasts with the laboratory procedure described in Section 3.2.2, where a defined sequence and controlled mixing times are strictly followed to ensure homogeneous dispersion of cement particles and consistent grout properties. Lumps of dry cement were observed in the system, indicating poor mixing. And different buckets of cement paste appeared to have different fluidities, also indicating inconsistent mixing and measuring throughout the grouting process.

When measuring out materials, the contractors measured 5 gallons of water in a bucket for each 94 lb. bag of cement. If a full 5 gallons is used per bag of cement, accurately, this would result in a 0.44 w/c. By not using a mass measurement system, however, the w/c ratio of 0.44 or 0.46 is likely approximate and may have been higher or lower. These discrepancies in mixing methods, environmental conditions, and handling logistics may contribute to variations in the grout's rheological properties, workability, and ultimately the mechanical and permeability performance of the cast elements, underscoring the importance of accounting for field-specific practices when interpreting and comparing test results.

Compressive Strength of Field Samples

A compression test was conducted on Type 1L-based grout samples at 3, 7, 14, 28, 56, and 90 days after casting. **Figure 8** illustrates the compressive strengths of field collected specimens and laboratory specimens. The laboratory data presented herein as "Type 1L-Lab" is the same data that is presented as "Type 1L in Section 4. It is repeated and relabeled here for convenience of comparison.

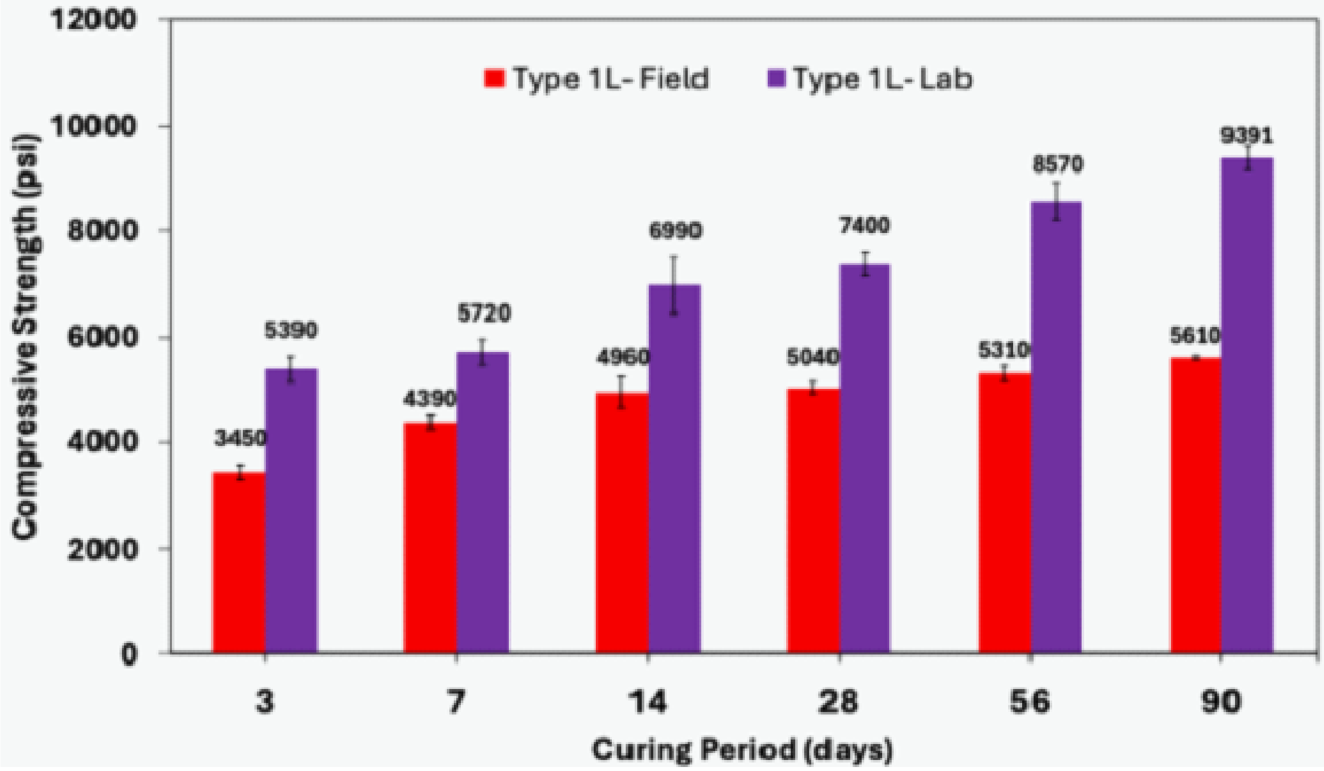

FIGURE 8. Compressive strength of Type 1L-based grout systems (field vs. lab)

As shown in Figure 8, a gradual increase of strength over time was exhibited by both field and laboratory produced grout specimens, but laboratory specimens consistently reached higher values. For early-age strength (28 days), lab specimens achieved an average strength of 7400 psi, while field specimens only reached 5040 psi, revealing reduction of 32%. For long-term strength (90 days), the strength was 5610 psi and 9391 psi, respectively, for field specimens and lab specimens, indicating higher strength gain for laboratory specimens. From 28 to 90 days, the strength increase was observed at 11% for field specimens, while it was about 27% for lab specimens.

Although field and laboratory specimens were cured under similar laboratory conditions after casting, the initial few hours are crucial for early hydration reactions and setting of fresh cement paste. The cooler ambient temperature (~18°C) during field casting may have delayed initial hydration compared to the controlled room temperature at 23±3°C, affecting the early development of microstructure. Moreover, the field and laboratory mixtures used cement from different manufacturers; variations in fineness, limestone content, and variable chemistry may influence hydration kinetics and ultimate strength, even at identical w/c ratios. Additional factors such as potential minor inconsistencies in field mixing, slight delays during transporting specimens to the laboratory, and uncontrolled vibration of the specimens during transportation could introduce variability, influencing early strength gain and long-term performance.

Practically, these results underscore the engineering challenge of replicating laboratory performance in field conditions. Even with laboratory curing after transport, differences in initial hydration conditions and cement properties led to a strength reduction of about 30-50% in the field specimens. It highlights the need for field-specific testing, careful mix design validation using actual materials and conditions, and potentially adjusting mixing protocols, or cement selection to ensure that field performance meets design requirements.

Despite these differences between the performance of the laboratory and field mixtures, it is important to note that the strength obtained by the field system is likely adequate for the application, reaching a strength above 4000 psi (normal-strength concrete) by 7 days after mixing. The current standard (N.J.A.C. 7:9D) does not list a minimum required strength for the grout systems.

Permeability Test

Permeability measurements carried out on field collected at 7, 14, and 28 days after mixing, the results of these measurements are presented in Table 10. Specimens were also taken to do a permeability measurement at 90-days after mixing, but the permeability measurement equipment in the laboratory suffered a malfunction, and at the time of this report, NJIT has been unable to repair it. The specimens were wrapped in plastic sheets and stored in the freezer to arrest ongoing hydration and maintain the paste integrity at 90 days until the machine is repaired. The 90-day results will be submitted in an amended report as soon as possible. Given the nature of hydration of cement, however, it should be expected that the permeability would remain similar or decrease between day 28 and day 90. Therefore, Day 28 results can be reliably used as an indication of quality for the material.

TABLE 10. Permeability results of field and laboratory produced specimens

Curing period (days)	Grout System	Permeability (cm/s)	
		Lab	Field
7	Type 1L cement-based	1.70E-10	7.43E-10
14		1.40E-10	12.60E-10
28		0.82E-10	7.88E-10

The development trend of permeability in Table 10 showed that the permeability remained relatively stable over time from 7 to 28 days in both laboratory and field specimens as hydration progressed and pore structures refined. In the laboratory, permeability declined steadily from 1.70×10^{-10} cm/s at 7 days to 0.82×10^{-10} cm/s at 28 days, indicating ongoing hydration and pore refinement. On the other hand, the permeability of field specimens was higher than the laboratory specimen's permeability at all ages, starting at 7.43×10^{-10} cm/s at 7 days and remaining elevated even at 28 days. The field system reached an average permeability of 7.88×10^{-10} cm/s at 28 days—about 10 times higher than the laboratory result at the same age. However, given that the order of magnitude of the permeability of both systems is the same (E-10), they should be regarded as relatively similar. Additionally, the permeability of field cast specimens were satisfactory and well below the maximum suggested limit of 1×10^{-7} cm/s per N.J.A.C. 7:9D-2.9 [2].

The differences in laboratory and field specimen results likely stem from a combination of material properties, inconsistent mixing, and early-age environmental effects. Although both systems use Type 1L cement and (nominally) the same w/c ratio, the field specimens utilized cement from a different manufacturer, likely varying in fineness or mineral composition. Such differences may affect the rate and extent of hydration and the development of the pore structure. Moreover, the early age temperatures that the field specimens were subjected to were lower than those of the laboratory. Those first hours are critical for pore network formation and slower hydration at lower temperatures (~18°C) may leave larger, more interconnected pores, explaining why the field specimens retained higher permeability throughout the curing period. Additionally, field mixing and handling conditions may have introduced subtle variations in actual water content or mixing efficiency, further influencing porosity and permeability.

Summary of Field Test Results

Results from testing of field specimens indicate that, despite discrepancies between the field results and laboratory results, Type 1L still performed adequately in the field. The discrepancies between results were likely caused by a variety of differences in the cement manufacturer, mixing procedures, and environmental conditions which can all impact the rate of hydration, strength gain, and permeability in cement-based systems. However, these results also indicate the importance of controlled mixing procedures for obtaining reliable and repeatable results.

Conclusion and Recommendation for Future Research and Application and Use by NJDEP

This study examined the viability of using Type1L cement in place of Type1 cement in well bore grouts in New Jersey. The results indicate that the Type1L systems performed similarly to their Type1 counterpart. In particular, the compressive strength and permeability of each system were of good quality, both from laboratory and field produced specimens. The permeability of each system met the requirements of N.J.A.C 7:9 for Well Construction and Maintenance, with permeabilities significantly lower than the maximum allowed permeability of $1x10^{-7}$cm/s.

Differences beyond standard testing variation were observed between the systems containing bentonite and those not containing bentonite. Bentonite systems, likely because of the high w/c, had significantly lower strengths and higher flowabilities, as well as being able to pass a charge more quickly through the systems. However, the permeability of the bentonite and non-bentonite systems was the same order of magnitude. For the systems without bentonite, the system containing limestone had slightly higher strengths and flowability, which is consistent with literature [56].

The systems with limestone also experienced higher levels of shrinkage when exposed to drying conditions, but when cured properly, shrinkage is not likely to be an issue of concern.

The performance of Type-1L specimens created during a field trial were observed to be worse than that of the Type-1L specimens created in the laboratory. The performance gap between laboratory and field specimens of Type-1L cement reflects the impact of uncontrolled variables such as inconsistent mixing, variations in cement source, and environmental conditions like lower ambient temperatures during placement. These factors can slow hydration and affect microstructural development, resulting in lower compressive strength and slightly higher permeability compared to laboratory-prepared samples.

Despite these reductions, the field specimens still achieved compressive strengths above 4,000 psi by seven days—a level generally considered sufficient for structural stability in well-sealing applications based on industry practice. The permeabilities were on the order of 10^{-10} cm/s, which is well below the regulatory limit of 1×10^{-7} cm/s specified in N.J.A.C. 7:9D for well-sealing applications. This demonstrates that, even under less controlled conditions, Type-1L cement provides sealing integrity to prevent fluid migration. These findings confirm compliance with the critical permeability requirement and indicate that the material performs reliably for its intended purpose when proper field practices are followed.

Based on the findings of this report, drilling contractors will observe only minor differences between well bore grouts using Type 1 and Type 1L cements. However, contractors should be sure to familiarize themselves with Type 1L cements before utilizing them in the field, as they may have slightly different flowabilities and setting times – resulting in different construction timing and procedures needed.

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