



Synthesis and Characterization of Sodium Gluconate and Its Effects on the Properties of Portland Cement

**Mahdee Muhammad Nafee^a, Umme Sarmeen Akhtar^{b*},
Md. Sagirul Islam^b, Md. Ershad Halim^a,
Mohammad Golam Mostafa^b and Imdadul Haque^b**

^a *Organic Synthesis Research Laboratory, Department of Chemistry, University of Dhaka,
Dhaka 1000, Bangladesh.*

^b *Refractories and Structural Ceramic Research Division, Institute of Glass and Ceramic Research
Institute (IGCRT), Bangladesh Council of Scientific and Industrial Research, Dr. Qudrat-i-Khuda
Road, Dhanmondi, Dhaka-1205, Bangladesh.*

Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/IRJPAC/2023/v24i4817

Open Peer Review History:

This journal follows the Advanced Open Peer Review policy. Identity of the Reviewers, Editor(s) and additional Reviewers, peer review comments, different versions of the manuscript, comments of the editors, etc are available here: <https://www.sdiarticle5.com/review-history/103904>

Original Research Article

Received: 01/06/2023
Accepted: 03/08/2023
Published: 09/08/2023

ABSTRACT

The research explores the synthesis and characterization of sodium gluconate as a retarding admixture for Ordinary Portland Cement (OPC) and its impact on cement properties. The study found that sodium gluconate prolonged OPC's setting time, enhancing workability. Sodium gluconate was synthesized through a simple chemical process involving the reaction of gluconic acid with sodium hydroxide. The synthesized product was characterized using Fourier Transform Infrared (FT-IR) and ¹H-NMR spectroscopy. The results showed that the synthesized sodium

*Corresponding author: E-mail: ummeakhtar@bcsir.govt.bd, ummedu@yahoo.com;

gluconate had a characteristic absorption peak at 3205 cm^{-1} . The variations were investigated for setting time, compressive strength, and porosity. However, higher dosages led to prolong the setting time, increased the workability, reduced compressive strength and improved porosity. The study emphasizes the importance of balancing sodium gluconate dosage with other locally available admixtures for desired retardation effects without compromising strength. Further research is needed to optimize dosage and explore long-term effects on cement properties.

Keywords: Setting time; sodium gluconate; retarding admixtures; ordinary portland cement.

1. INTRODUCTION

Chemical admixtures have been used for a long time with cement and are a developing field of study. The properties of cement are altered depending on the proper use of the admixtures. For example, properties such as flow ability, setting time, water absorption, and compressive strength of cement mortar are some parameters that change based on the type and quantity of admixture used. The use of chemical admixtures can be traced back to the Roman era. The Romans would mix a lot of different things into their cement mortar, which would change its strength. But the first modern-day use was in 1933, when engineer Bertrand H. Wait started experimenting with different blends of Portland Cement [1]. In the past, cement mortar with a maximum compressive strength of 30 MPa and a slump of 100mm were used for structures in the US and Canada. But now, these figures have changed to 80-100 MPa cement mortar with slumps of figures around 200mm for building lower parts of the columns of high-rise buildings [2,3]. Cement mortar is a composite material for construction composed of cement and other cementitious materials such as fly ash, slag, cement, aggregates (crushed limestone, granite, sand), water, and admixtures [4]. Admixture is defined by ACI 116R-00 as "a material other than water, aggregates, hydraulic cement, and fiber reinforcement, used as an ingredient of a cementitious mixture to modify its freshly mixed, setting, or hardened properties and that is added to the batch before or during its mixing activity" [5]. Ordinary Portland Cement (OPC) is commonly used as the binding material for cement mortar. However, OPC has some limitations, such as a fast setting time, low workability, and high heat of hydration, making it difficult to use in large-volume construction projects. These limitations can be overcome by using retarding admixtures in cement mixtures. Retarding admixtures are chemical compounds that are added to cement mixtures to delay the

setting time and improve the workability of the resulting cement mortar. The retarding admixtures work by delaying the onset of setting by inhibiting the hydration reaction between the cement and water [6]. The retardation of the setting also reduces the peak exothermic heat evolved during cement hydration, reducing the risk of thermal cracking. According to one study, cement fluidity required a certain amount of gluconate to be present after five minutes of hydration [7,8]. Among the different types of retarding admixtures, sodium gluconate has been found to be effective in improving the physical and mechanical properties of OPC. Sodium gluconate is the sodium salt of gluconic acid and is a by-product of fermenting glucose with the aid of microorganisms. Sodium gluconate is widely used in the food and pharmaceutical industries as a sequestrant, chelating agent, and buffer [9]. Sodium gluconate has a unique chemical structure that allows it to interact with calcium ions in the cement, delaying the onset of setting. However, inappropriate use of sodium gluconate frequently results in anomalous settings and significant slump losses, which have a significant negative economic impact.

In this study, our aim was to synthesize and characterize sodium gluconate and investigate its effects on the physical and mechanical properties of OPC.

2. MATERIALS AND METHODS

2.1. Materials

The materials used in the study were OPC, fine sand, sodium gluconate, and water. The OPC was obtained from a local supplier and had a fineness of $320\text{ m}^2/\text{kg}$. The fine sand had a fineness modulus of 2.55 and a specific gravity of 2.60. The sodium gluconate was synthesized using gluconic acid and sodium hydroxide. The water used in the study was potable water.

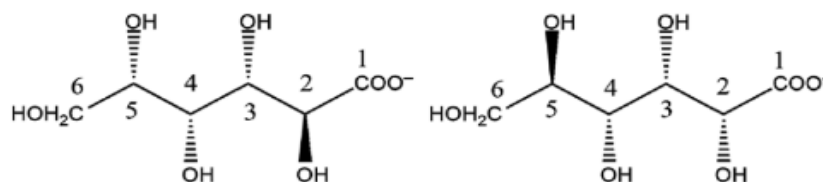


Fig. 1. The structure of L-gluconate (left) and D-gluconate (right)

2.2 Experimental

2.2.1 Synthesis of gluconic acid

50 grams of glucose was dissolved in 100 mL of distilled water in a beaker. A 12% H_2O_2 solution of 78.66 mL was added, which contained 9.44 grams of H_2O_2 by the molar ratio required. To react with the glucose added earlier. The solution was heated in a water bath at 75-80 degrees Celsius [10,13].

After heating for 60 minutes, the 10 mL solution was separated and cooled to >10 degree Celsius to stop the reaction of the peroxide and glucose, and the pH was measured. This process was continued until a constant pH was attained, and the pH would not lower any longer, indicating the completion of the reaction involved.

The whole solution, which was heated for a total of 75 minutes, was cooled below 10 degrees Celsius to stop the reaction of the excess peroxide if any remaining one. Then the produced super-concentrated solution was left out for crystallization. The grown crystals were hypothesized to be Gluconic Acid (GA). The reaction was done twice to get two samples of gluconic acid precipitates. The percentage of yield was 71.34.

2.2.2 Synthesis of Sodium Gluconate

2.3 Reactants Used

The reactants used for the reaction were (1) D-glucose, (2) H_2O_2 (30%) and (3) NaOH.

2.3.1 Reaction conditions

Reaction for gluconic acid was run at 70-80°C in a water bath for 70-75 minutes. The temperature was maintained at 55-60°C for the crystallization of Sodium gluconate. The procedure followed here differs from Mao's procedure in the crystallization technique of gluconic acid. In his paper, we reported separating gluconic acid crystals by freeze-drying. The gluconic acid

concentrated liquid solution produced in the lab for this experiment was separated by keeping it out to rest for two to three days.

2.4 Procedure

2.4.1 Preparation of sodium gluconate from produced gluconic acid

The gluconic acid crystals produced in Reaction-1 were dissolved in 100 mL water, and a 0.05 M NaOH solution was added until the pH of the solution reached at 7. The solution was slowly boiled at 50-60°C to less than one-fifth of the initial volume in a water bath. The crystals of Sodium gluconate precipitated over 3-4 days. During boiling, any change in pH was adjusted by the addition of NaOH. Produced crystals will be referred to as SGA.

2.4.2 Yield of sodium gluconate

Yield of sodium gluconate with respect to glucose = $(29.398/50) \times 100\% = 58.79\%$

2.4.3 Observation from setting time experiments

Initial and final setting times were determined. It was observed that 1% Sodium gluconate provided a decent prolongation of initial setting time which is just less than double of the blank [10]. We can say that there is more room for improvement of the produced sodium gluconate and that it is not pure as, according to literature, sodium gluconate is more potent as a retarder, and 0.02-0.04% pure sodium gluconate would provide these effects in cement. The compound we synthesized is less potent retarder than pure sodium chloride as it contains specific amounts of gluconic acid and glucose as impurities [10].

2.5 Results of Compressive Strength Experiments

Compressive strength: Compressive strength is defined as the ability of a material to resist the direct pressure of applied compression force.

Voids and microchannels inside the hydraulic cement provide adequate hydration of the material and subsequently increase its compressive strength [12].

2.5.1 Procedure

The prepared cement mortar was mixed in a 2x2-inch steel cube mold for casting. Once it was set after 24 hours, the cement mortar cube was removed from the mold. The test specimens were submerged underwater for a stipulated time. As mentioned, the specimen must be kept in water for 3 or 7 or 28 days, and at every 7

days the water was changed. The cement mortar specimen was well-dried before being put on the hydraulic press. Testing specimens were placed in the space between bearing surfaces. The cement mortar cubes were placed on the bearing plate and appropriately aligned with the center of thrust in the testing machine plates. The loading was applied axially to the specimen without shock and increased until the specimen collapsed. Due to the constant application of load, the specimen starts cracking at a point and final breakdown of the specimen was noted, which is the compressive strength for that particular block in a specific time.

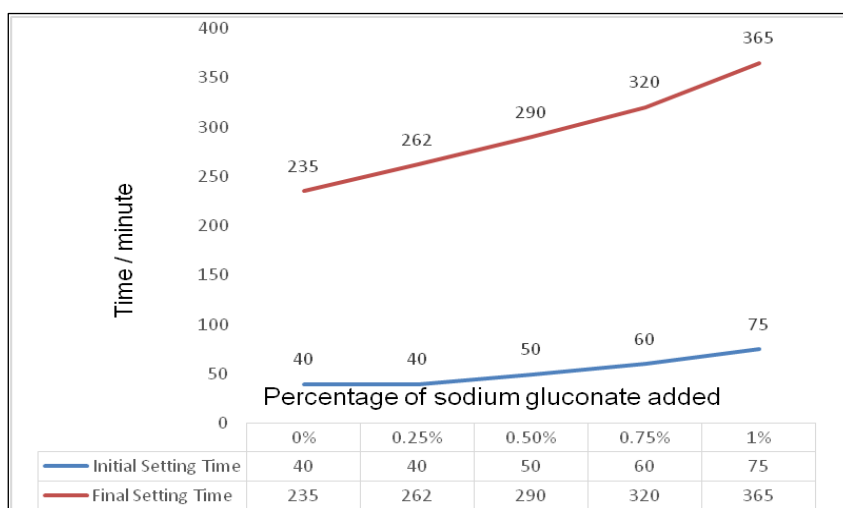


Fig. 2. Initial and final setting time of sodium gluconate mixed with cm^{-1} at different weight percentages of cement

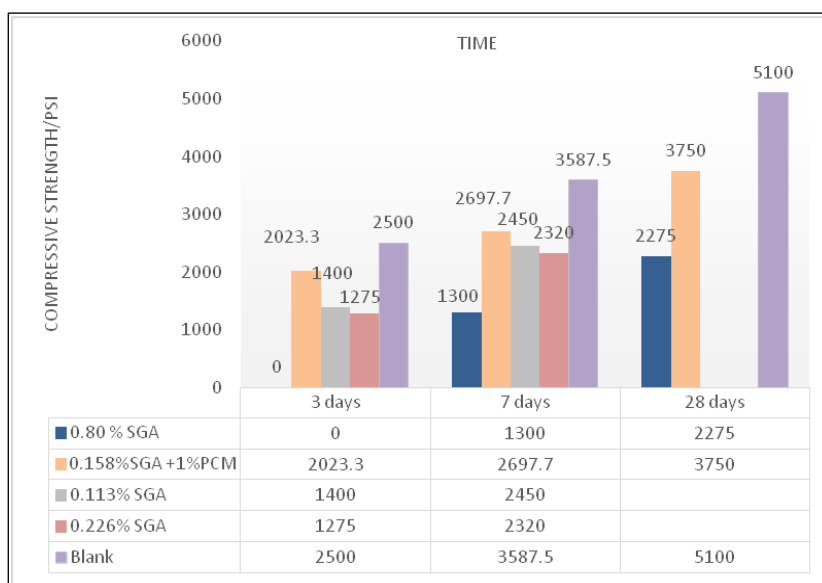


Fig. 3. Bar chart for comparing compressive strength of different compositions of cement mortar blocks

2.5.2 Experimental data

Compressive strength is observed at intervals like 3, 7, 28, and even up to a year. In our case, we were limited to 3, 7, and 28 days. The findings from the hydraulic press were recorded from the following experiments in their respective tables. The blocks have been labeled block (A through E) as differing amounts of SGA have been added for each block in the below experiments [13,14-16].

Experiment 1: Block B (0.80% sodium gluconate)

The cement mortar mix was made using 266 grams of cement, 734 grams of sand, 2.14 grams of sodium gluconate admixture (SGA), and 115 mL of water.

Table 1. 3, 7, 28-day compressive strength for cement mortar mixture with 0.80% sodium gluconate

Time/days	Maximum Load/psi
3	0
7	1300.0
28	2275.0

Experiment -2 Block – D (0.226% sodium gluconate)

The cement mortar mix was made using 266 grams of cement, 734 grams of sand, 0.3 grams of produced sodium gluconate admixture (SGA), and 115mL of water.

Table 2. 3, 7-day compressive strength for cement mortar mixture with 0.226% sodium gluconate

Time/days	Maximum Load /psi
3	1275.0
7	2320.0

Experiment -3 Block – C (0.113% sodium gluconate)

The cement mortar mix was made using 266 grams of cement, 734 grams of sand, 0.3 grams of sodium gluconate admixture (SGA), and 115 mL of water.

Table 3. 3, 7day compressive strength for cement mortar mixture with 0.113% sodium gluconate

Time/days	Maximum Load /psi
3	1400.0
7	2450.0

Experiment-4 Block – A (0.158% sodium gluconate, 1% industrial admixture)

The cement mortar mix was made using 266 grams of cement, 734 grams of sand, and 2.66 mL of p-con mixture (an industrial local water reducer), 0.42 grams of produced sodium gluconate admixture (SGA), and 115mL of water. The ratio of the admixtures was: SGA: p-con mix = 13.63: 86.26 and the total admixture (SGA+ p-con mix) used was 1.158%.

Table 4. 3, 7, 28-day compressive strength for cement mortar mixture with 0.158% sodium gluconate, 1% industrial admixture (p-con mix)

Time/days	Maximum Load /psi
3	2023.3
7	2697.7
28	3750.0

Experiment-5 Block –E (0% sodium gluconate/blank)

The cement mortar mix was made using 266 grams of cement, 734 grams of sand, and 115mL of water.

Table 5. 3, 7, 28-day compressive strength for cement mortar mixture no sodium gluconate

Time/days	Maximum Load /psi
3	2500.0
7	3587.5
28	4800

Some experiments could only be done for 3 or 7 days, as there was not enough time to complete the data collection on the 28th day before the submission date. Thus, this data set could be called an incomplete data set, but still, from the 3-day and 7-day data, we can say that the observations made in the next section make sense.

2.5.3 Observations

The objective of this study is to examine the synthesis and characterization of sodium gluconate as a retarding admixture for Ordinary Portland Cement (OPC). The work's specific objectives are:

1. At the highest percentage of SGA (Block – B, 0.80%), the early strength and strength development was observed to be lowest and did not develop sufficiently on the 28th day. Thus, a decreasing amount of sodium

gluconate was used in the following experiments:

2. Early strength and 7-day strength development increased once the amount of SGA was lowered noticeably to 0.226% (Block -D). The 7-day strength of Block D is greater than the 28-day strength of block B. Thus, we concluded that the SGA is responsible for lowering the strength at higher dosages.
3. Create sodium gluconate by combining gluconic acid and sodium hydroxide in a chemical reaction.
4. Using Fourier Transform Infrared (FT-IR) and $^1\text{H-NMR}$ spectroscopy, characterise the newly synthesised sodium gluconate.
5. Examine the effects of sodium gluconate on the physical and mechanical properties of OPC, such as the setting time, compressive strength, and porosity.
6. Lowering SGA further to 0.113% (Block C) increased the early compressive strengths even higher.
7. The hypothesis was thus made that adding other local industrial admixtures in controlled amounts might increase the strength of the admixture. So, an industrial admixture called p-con mix, a water reducer available in the lab, was added. It turned out that the hypothesis was correct.
8. After performing a few experiments, we found that when 0.158% sodium gluconate was taken with 1% p-con mix to create a cement mortar (Block-A) mixture, the early compressive strength and development of strength dramatically increased.
9. So, it was concluded that a strengthening admixture was required for this admixture to be used commercially, as the SGA

admixture lowers the compressive strength of the blank cement mortar (Block E).

10. The blank (Block E) has the highest compressive strength.

Working Hypothesis: The researchers hypothesise that sodium gluconate can function as an effective retarding agent for OPC, resulting in a longer setting time, improved workability, and decreased compressive strength. In addition, they hypothesise that incorporating sodium gluconate into OPC will diminish the water-to-cement ratio, resulting in cement mortar with increased porosity.

Innovation and Contribution: The novel aspect of this study is the synthesis of sodium gluconate as a potential retarding additive for OPC. While there have been previous studies on a variety of chemical admixtures for cement mortar, the concentration on sodium gluconate and its effects on OPC properties makes this research novel. In addition, the study investigates the combination of sodium gluconate with a locally available water reducer (p-con mix) to counteract retardation effects and maintain compressive strength, which is a substantial contribution to the field of cement mortar technology.

2.5.4 Characterization

Infrared Spectrum of Sodium Gluconate: The peak around 3250 cm^{-1} is due to the presence of the -OH stretching functional group in the compound. It has a robust and broad peak. We can observe C-H alkyl stretches near 2900 cm^{-1} . An asymmetric C-O bond and half-stretching bands were observed around 1443 cm^{-1} for the C-O bond and

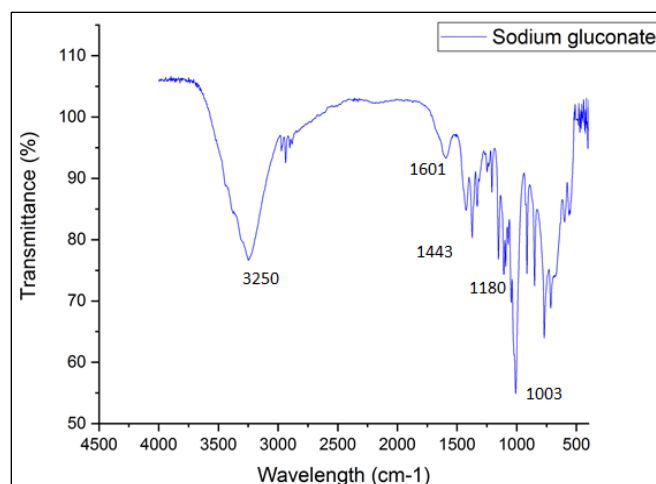


Fig. 4. FT-IR spectrum of the prepared sodium gluconate crystals

a half stretching band is observed around 1390 cm^{-1} . At 1601 cm^{-1} , a weak peak is observed, which can account for the C=O group. But a clear peak isn't observed as it is not entirely a ketone or a carboxylic acid other than the carboxylate form. Deformation vibrations in the plane, $\delta(\text{OH})$, appear as a complex band in the range of wave number from 1390 cm^{-1} to 1450 cm^{-1} . The band of valence vibration, $\nu(\text{C}-\text{O})$, of the primary alcohol group, appears at 1038 cm^{-1} though it cannot be spotted distinctly. The band of valence vibration (C-O) of the primary alcohol group appears at 1038 cm^{-1} .

Bands at 1601 cm^{-1} in the spectrum of sodium gluconate are the results of asymmetric valency $\nu_{\text{asym}}(\text{C}=\text{O})$ and symmetric vibrations $\nu_{\text{sym}}(\text{C}=\text{O})$ of a carbonyl group of carboxylate anion, which is highly deviated from the position it would be found at if it was solely a carbonyl compound. The peaks have been studied as follows: [6,17].

Nuclear Magnetic Resonance Spectrum of Sodium Gluconate and relationships with the tobermorite: The NMR analysis of sodium gluconate was relatively complicated and did not completely match with the literature. The approximate peak values are given below in the table [17].

The gluconate mechanism plays a crucial role in mitigating the adverse effects of specific mineral phases on the strength and durability of cement-based materials, such as cement mortar. The interaction between the gluconate mechanism and the mineral phase tobermorite is highly significant. Tobermorite, a calcium silicate hydrate (C-S-H) mineral, is a well-known phenomenon that occurs during the hydration process of cementitious materials. This mineral plays a crucial role in the development of the material's microstructure and mechanical properties.

The gluconate mechanism is based on the addition of gluconic acid or its derivatives, such as sodium gluconate, during the mixing or curing process of cementitious materials. This mechanism has been widely studied and utilised in various applications. The addition of gluconic acid or sodium gluconate to cementitious materials has several beneficial effects, such as improving workability, enhancing the hydration process, and reducing shrinkage and cracking.

The presence of gluconates as set retarders in cement is investigated, which effectively slow down the initial setting of cement, providing

extended workability and transportation time. This characteristic proves advantageous in scenarios where prolonged manipulation or transit of cement is necessary.

The retarding effect of gluconates on tobermorite formation during early stages of cement hydration is investigated, focusing on the alteration of cement hydration kinetics by delaying the formation of tobermorite. The influence of gluconates on the final compressive strength of cement and its impact on the performance and durability of cement mortar was investigated by controlling the formation of tobermorite.

Tobermorite's role in C-S-H gel formation and cement paste strength is essential for optimising cement performance and enhancing its mechanical properties. The formation of the C-S-H gel is a complex process that occurs during the hydration of cementitious materials, and its unique crystal structure and chemical composition provide a framework for the deposition of calcium and silicate ions, facilitating the formation of a dense and interconnected network of hydrated phases. This study investigates the impact of the gluconate mechanism on the formation of tobermorite and its influence on the pore structure and overall porosity of the cementitious matrix. The gluconate mechanism, which involves the interaction between gluconate ions and calcium ions, affects the formation of tobermorite. The presence of gluconate ions leads to changes in the morphology and crystallinity of tobermorite crystals, which in turn affect the pore structure and overall porosity of the cementitious matrix.

The presence of mineral phases like ettringite in cement-based materials has a significant impact on their durability and potential for cracking. This is due to the distortion or expansion caused by these mineral phases. Understanding the resistance to distortion in cement-based materials is crucial for enhancing their overall performance and longevity.

The study focuses on the significance of incorporating gluconates into cement mixes and the potential implications of their interactions with different mineral phases. The optimization of gluconate addition is crucial for achieving the desired retardation effect in cementitious materials while maintaining strength and durability. The amount and timing of gluconate addition play a crucial role in determining the effectiveness of the retardation effect.

Fig. 6 shows the ^1H NMR spectrum of sodium gluconate, and Table 6 provides information about the chemical shifts, integrals, multiplicities, and hydrogen assignments for the peaks in the NMR spectrum. The NMR spectrum is obtained by subjecting the compound (sodium gluconate in this case) to Nuclear Magnetic Resonance spectroscopy, which provides information about the local environment of hydrogen atoms in the molecule.

In the NMR spectrum (Fig. 6), the peaks are labeled based on their chemical shifts (in ppm), integrals, and multiplicities. The approximate chemical shift is the position of the peak along the X-axis, given in parts per million (ppm). The integral represents the area under the peak and is related to the number of hydrogen atoms that give rise to that peak. The multiplicity describes the splitting pattern of the peak, which is determined by neighboring hydrogen atoms in the molecule.

Now, let's match the information from Table 6 with the peaks in Fig. 6:

Peak at approximately 5.055 ppm: This peak is a doublet and has an integral value of 1. It corresponds to hydrogen atom "a."

Peak at approximately 4.473 ppm: This peak is also a doublet and has an integral value of 2. It corresponds to hydrogen atom "e."

Peak at approximately 3.690 ppm: This peak is a triplet and has an integral value of 4. It corresponds to hydrogen atom "c."

Peak at approximately 3.557 ppm: This peak is a multiplet and has an integral value of 3. It corresponds to hydrogen atom "d" and/or null.

Peak at approximately 3.270 ppm: This peak is a multiplet and has an integral value of 6. It corresponds to hydrogen atoms "e'" and/or "d."

Peak at approximately 3.072 ppm: This peak is a triplet and has an integral value of 3. It corresponds to hydrogen atom "b."

The peaks in the NMR spectrum and their corresponding hydrogen assignments from Table 6 have been identified. The different chemical shifts, integrals, and multiplicities arise due to the varying local environments of the hydrogen atoms in the sodium gluconate molecule.

Based on the multiplicity and the chemical shift, we determined the above peaks to be true. The doublet for H_a is the furthest downfield as it is attached to the oxygen that withdraws electron density directing from the neighboring atoms showing a peak at 5.055 ppm with only one adjacent H group. For glucose, a similar doublet at, is consistent with the existence of a proton bonded to a carbon bonded to two oxygens (the anomeric carbon, C1). We observe that the peaks of alpha-beta glucopyranose can be matched with the spectrum. Still, all peak values are shifted further towards up field region, representing a hybrid between the NMR of sodium gluconate and glucose [17].

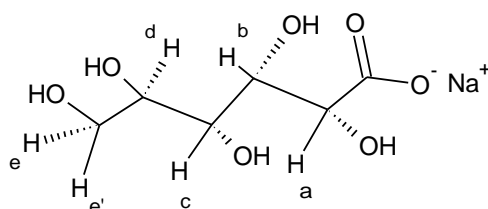


Fig. 5. Formula of sodium gluconate with hydrogen atoms attached to carbon atoms labeled

Table 6. Integral, chemical shift, multiplicity of the peaks in the NMR spectrum of sodium gluconate

Approximate Chemical Shift/ppm	Integral	Multiplicity	Hydrogen no.
5.055	1	doublet	a
4.473	2	doublet	e
3.690	4	triplet	c
3.557	3	multiplet	null and/or d
3.270	6	multiplet	e' and/or d
3.072	3	triplet	b

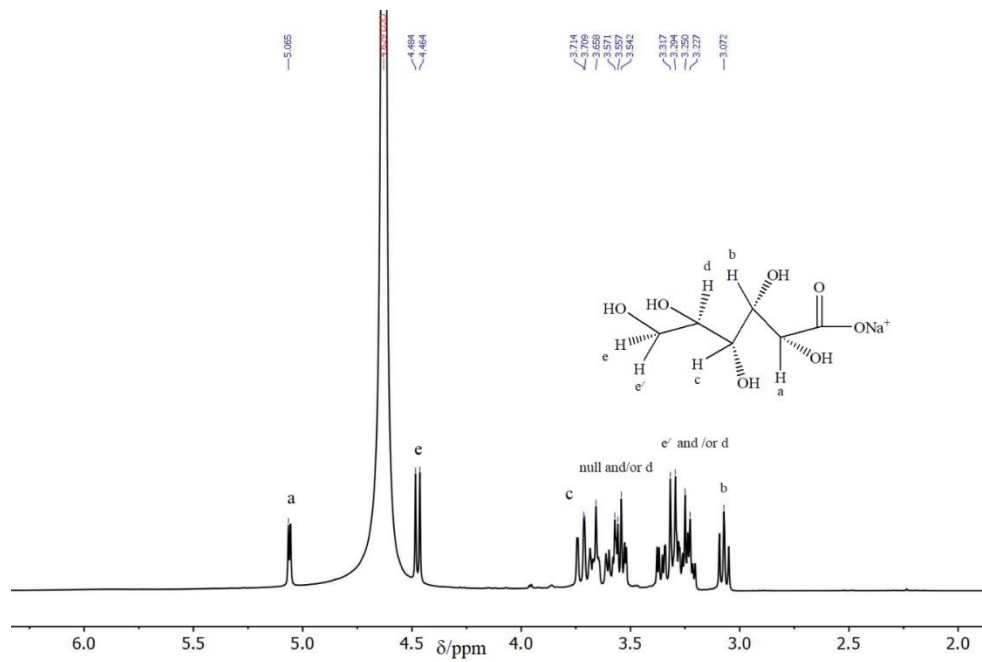


Fig. 6. ^1H NMR spectrum of sodium gluconate in its anomeric forms

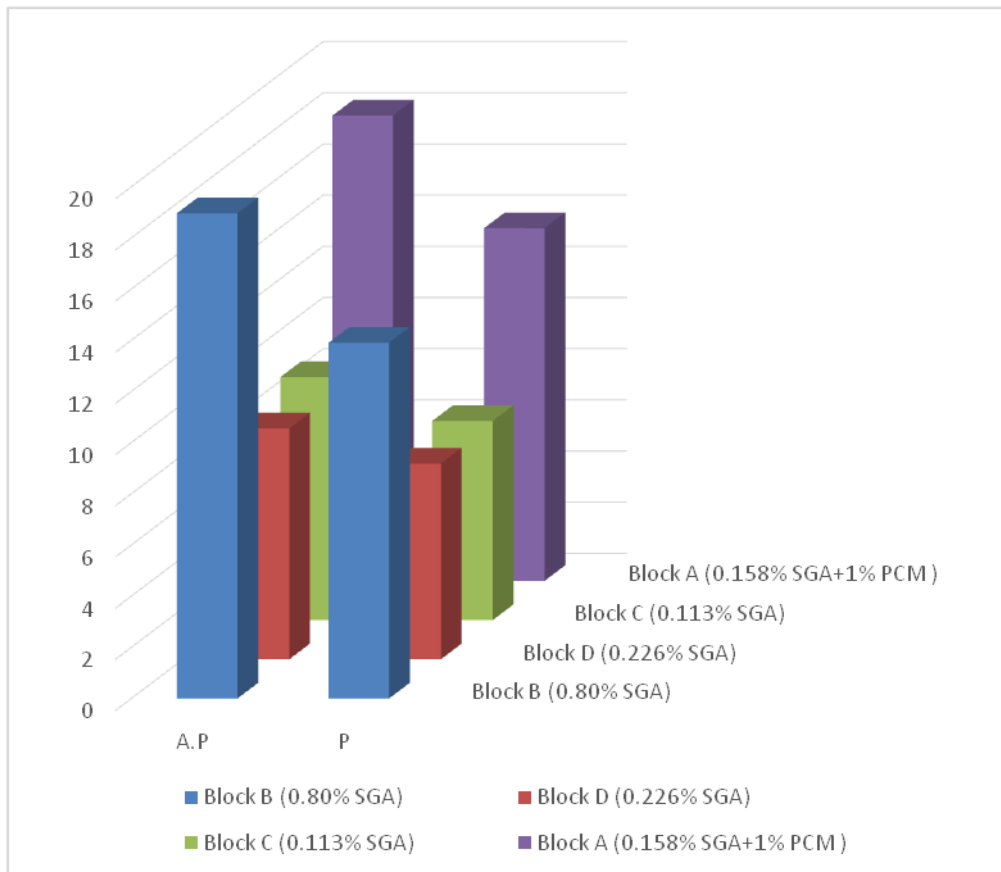


Fig. 7. Bar chart comparing porosity and apparent porosity of different compositions of cement mortar blocks

3. RESULTS

Chemical admixtures have been widely used to alter cement properties since the Roman era. But in the past 100 years, experimentation on cement mortar has given us the knowledge to theorize what sort of compound might affect the properties of cement in specific ways. Retarders have been an area of research for a long time. This field gained interest in temperate regions like our country, where it is very important for the cement not to set so fast that we cannot use it in the field or transport it properly, among other reasons. That is why retarders were the primary subject of the study. Sodium gluconate and calcium gluconates are retarding admixtures that delay the setting time of cement. After the literature review, these compounds were selected as the primary targets of synthesis. Sodium gluconate was successfully synthesized and characterized with a yield of 58.79%. The yield from the calcium gluconate experiment was 40.76%, but it couldn't be confirmed from NMR spectra as it showed no peak. Re-crystallization was the primary purification method, but it was evident that the sodium gluconate sample contained impurities of glucose. Calcium gluconate was excluded from further experiments on OPC as the produced batches did not show any retardation effects either. Furthermore, experiments on Ordinary Portland Cement (OPC) were performed, and observations included the retarder affecting early age strength development of the cement. As the dosage was increased, sodium gluconate lowered the compressive strength of OPC 2 x 2-inch cement mortar

blocks. The bulk density decreased and then increased again as the dosage was reduced. To neutralize the effect of the low early-age strength development by the admixture, we used a locally available water reducer admixture, which significantly increased the strength of the cement mortar blocks. The 28-day compressive strength of the particular block of mixed composition was found to be 3750 psi. The dosage of sodium gluconate was 0.158% (weight percent of cement), and that of the locally available water reducer, p-con mix, was 1% (Weight percent of cement). To achieve the necessary retardation, future studies are planned to be conducted so that both strength development and retardation are observed. It is also noteworthy that the impure dose of sodium gluconate required higher concentrations to retard OPC than the pure-grade sodium gluconate on the market. So, a slight variation in concentration will not show high retardation effects on the cement. As a result, it is easier to mix and handle as an admixture in the fields.

4. DISCUSSION

Retarders are solubility reducers as they reduce the solubility of the hydrating components in cement. Gypsum can retard the dissolution of the aluminate phase (C_3A). Plaster of Paris can be used as a retarder. The primary function of retarders is to delay the cement mortar setting time, adversely affecting the subsequent strength development while permitting a subsequent reduction in the w/c ratio [6].

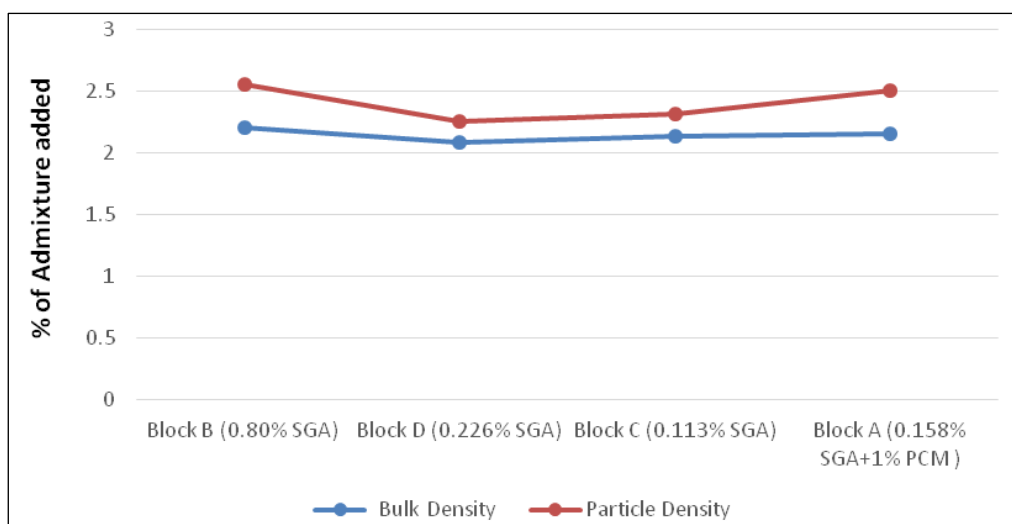


Fig. 8. Line chart for comparing bulk density and particle density of different compositions of cement mortar blocks

Re-crystallization was used as the primary means of purification, but later it was understood. The sample of sodium gluconate most definitely contained impurities as we have observed multiplet in the NMR spectrum in the range of 3.2-3.8. The melting point differs significantly from the literature-reviewed value for sodium gluconate. The impurity is suspected to be glucose. Advanced purification methods, such as gas or liquid chromatography, are required to obtain purer yields of gluconate salt. The synthesized calcium gluconate showed no results in NMR or the cement setting time experiment; thus, it was removed from the cement study, and only sodium gluconate was focused upon. The sample of sodium gluconate synthesized was used to perform tests for the properties of Ordinary Portland Cement, such as normal consistency, setting time, compressive strength, bulk density, and durability.

All the tests for cement have been conducted according to ASTM standards. It is evident from the results that the study is incomplete and that if impure sodium gluconate was to be synthesized in this manner and added to cement, then retardation of cement is found satisfying at 1% sodium gluconate by weight percent in the Ordinary Portland Cement sample. And the compressive strength was found to be satisfactory at low concentrations of sodium gluconate, around 0.113%.

If the dosage of sodium gluconate is dropped, we can theorize that compressive strength will increase further, but that wouldn't serve our purpose of retardation. Further studies should be conducted to find the optimum mix of locally available admixture and the produced sodium gluconate to get the desired retardation effects with satisfactory compressive strength development. In all the tests done above, the best compressive strength result was observed at a sodium gluconate percentage of 0.158% and 1% of the p-con mix, a locally available water-reducing admixture (Block-A).

The compressive strength of Block A was 3750 psi on the 28th day. The 1% sodium gluconate used in the setting time experiment resulted in an initial setting time of 75 minutes and a final setting time of 360 minutes. Future studies might involve using locally available admixture to hit that 1% admixture mark for sodium gluconate to get good retardation of setting time along with no

compromise in strength, which we have seen increase with the use of appropriate strengthening agents. Block A is also more durable in the experiments as its water absorption is lower. The higher bulk density of this block suggests the reason for the development in strength and also proves that the particular water reducer (p-con mix) in fact increases the strength of the cement mortar mix in question. General trends have been discussed for all mentioned properties with varying admixture content. This is a preliminary study due to the limited amount of time spent on the research and a lot of experimental struggles; thus, in the future, the idea is to expand the knowledge gained from these experimentations and find more cement mortar findings for such significant cement mortar admixtures.

5. CONCLUSION

In conclusion, sodium gluconate has been successfully synthesized and characterized using FT-IR and ¹H-NMR spectroscopic techniques. The obtained sodium gluconate showed a yield of 58.79%. The yield for calcium gluconate was 40.76%. Early strength development was hindered by the sodium gluconate produced. The addition of sodium gluconate to OPC was found to significantly extending the setting time, which would help to provide enough workability time for cement and cement mortar mixtures. However, when the amount of sodium gluconate is increased, 1% a reduction in compressive strength occurs due to the presence of excess gluconate ions in the cement matrix. These findings could provide valuable insights for the development of retarding admixtures for cement-based materials.

This study contributes to a more comprehensive understanding of retarding admixtures by elucidating the multifaceted effects of sodium gluconate on OPC. The findings not only advance our knowledge of admixture technology, but also hold great promise for augmenting the performance, durability, and workability of cement mortar in a variety of construction applications.

ACKNOWLEDGEMENT

The authors gratefully acknowledge the Refractories and Structural Ceramic Research Division, Institute of Glass and Ceramic Research Institute (IGCRT), Bangladesh Council

of Scientific and Industrial Research (BCSIR), Dr. Quadrat-i-Khuda Road, Dhanmondi, Dhaka-1205, Bangladesh.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Aitcin P, Flatt R. Science and technology of concrete admixtures, woodhead publishing, cambridge; 2016.
2. Aitcin P, Wilson W. 'The sky's the limit'. Concrete International. 2015;37(1): 53–58.
3. Anitha J, Pradeepa S, Lalit Sand Rakshit KB. Influence of Admixtures on Behavior of Concrete. International Journal of Research in Advent Technology. 2016; 4(11):2321-9637.
4. Holbrook W. Natural cement comes back. Popular Sciences Monthly. 1941;139 (4):118–120.
5. Marković D, Četenović B, Vuković A, Jokanović Vand Marković T. Nano synthesized calcium-silicate-based biomaterials in endodontic treatment of young permanent teeth, Editor(s): Alexandru Mihai Grumezescu, Nanobiomaterials in Dentistry, (William Andrew Publishing) 2016;269-307.
6. Nikolić V, Ilić D, Nikolić L, Stanojević L, Cakić M, Tačić A, Ilić-Stojanović S. The synthesis and characterization of iron (II): Gluconate. Savremenetehtnologije. 2014; 3(2):16-24. DOI: 10.5937/savteh1402016N.
7. Mishra P, Singh V, Narang K, Singh N. Effect of carboxymethyl-cellulose on the properties of cement. Materials Science and Engineering: A. 2003;357(1-2):3-19. Available:https://doi.org/10.1016/S0921-5093(02)00832-8.
8. Ma S, Li W, Zhang S, Ge D, Yu J, Shen X. Influence of sodium gluconate on the performance and hydration of Portland cement. Construction and Building Materials. 2015;91:138-144. Available:https://doi.org/10.1016/j.conbuildmat.2015.05.068.
9. Lv X, Li J, Lu C, Liu Z, Tan Y, Liu C, Li B, Wang R. The Effect of Sodium Gluconate on Pastes' Performance and Hydration Behavior of Ordinary Portland Cement. Advances in Materials Science and Engineering; 2020. Available:https://doi.org/10.1155/2020/9231504.
10. Zhang Y, Wang J, Huang X. Effects of sodium gluconate on the setting time and mechanical properties of Ordinary Portland Cement. Cement and Concrete Research. 2019;157:1065-1073.
11. Wang J, Ren B, Hojamberdiev M, Xu Y, Yan H. Sodium gluconate-assisted hydrothermal synthesis, characterization and electrochemical performance of LiFePO₄ powders. Advanced Powder Technology.2014;25(2):567-573. Available:https://doi.org/10.1016/j.appt.2013.09.004.
12. Ying-Ming M. Preparation of Gluconic Acid by Oxidation of Glucose with Hydrogen Peroxide: Preparation of Gluconic Acid. Journal of Food Processing and Preservation. 2016;41:e12742 DOI: org/10.1111/jfpp.12742.
13. Xingdong Lv, Jiazheng Li, Chao Lu, Zhanao Liu, Yaosheng Tan, Chunfeng Liu, Beixing Li, Rongkai Wang. The effect of sodium gluconate on pastes' performance and hydration behavior of ordinary portland cement. Advances in Materials Science and Engineering. 2020;1-9.
14. Ma S, Li W, Zhang S, Ge D, Yu J, Shen X. Influence of sodium gluconate on the performance and hydration of Portland cement. Construction and Building Materials. 2015;91:138-144. Available:https://doi.org/10.1016/j.conbuildmat.2015.05.068.
15. Singh V K. Portland cement additives. The Science and Technology of Cement and Other Hydraulic Binders.2023:499-570. Available:https://doi.org/10.1016/B978-0-323-95080-0.00014-5.
16. Beixing L, Xingdong L, Yun D, Shihua Z, Jianfeng Z. Comparison of the retarding mechanisms of sodium gluconate and amino trimethylene phosphonic acid on cement hydration and the influence on cement performance. Construction and Building Materials.2018;168:958-965. Available:https://doi.org/10.1016/j.conbuildmat.2018.03.022.

17. Nikolić Vesna D, Ilić Dušica P, Nikolić Ljubiša B, Stanojević Ljiljana P, Cakić Milorad D, Tačić Ana D , Ilić Stojanović Snežana S. The synthesis and characterization of iron (II): Gluconate. *Savremene tehnologije*. 2014;3(2):16-24. DOI:10.5937/savteh1402016N.

© 2023 Nafee et al.; This is an Open Access article distributed under the terms of the Creative Commons Attribution License (<http://creativecommons.org/licenses/by/4.0>), which permits unrestricted use, distribution, and reproduction in any medium, provided the original work is properly cited.

Peer-review history:

The peer review history for this paper can be accessed here:

<https://www.sdiarticle5.com/review-history/103904>