

Effect of Biodegradable Dextrin LU-1400-2 on Selected Properties of Cement Mortars

Marta Sybis^{1*)}

^{1*)} Department of Construction and Geoengineering, Poznan University of Life Sciences, Piatkowska 94 E, 60-649 Poznan, Poland; e-mail: marta.sybis@up.poznan.pl; ORCID: https://orcid.org/0000-0002-0032-6313

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Abstract

This paper presents the results of a study on the effect of fully biodegradable modified starch in the form of LU-1400-2 dextrin (denoted d2) and two commercial plasticizers P1 and P2 on selected properties of cement mortars. The studied cement mortar was enriched with 0.25%, 0.30%, 0.35%, and 0.40% d2 dextrin, relative to the weight of the cement. The tests carried out indicate that the addition of dextrin to cement mortars results in an increase in strength (a 9% increase was observed with 0.25% dextrin) and liquefaction of the mixtures. However, commercial plasticizers were found to decrease compressive strength. Starch derivatives are a type of natural plasticizer that is more environmentally sustainable than other types. They are produced from renewable sources, such as plants, and their production generates less waste and involves low greenhouse gas emissions. They are produced from renewable sources suggest that dextrins have great potential as natural plasticizers. The increasing popularity of natural plasticizers in the chemical and construction industries is due to a growing interest in sustainability and concern about the harmful effects of traditional chemical plasticizers.

Keywords: starch, rheology, cement mortar, compressive strength

1. Introduction

Construction is one of the world's primary sources of carbon dioxide emissions, accounting for approximately 40% of total CO2 emissions. This result consists of emissions from both the construction process and the operation of buildings. The construction process, which includes the extraction of raw materials, the production of building materials, transport, and the construction work itself, generates a considerable amount of carbon dioxide. The most significant contributor is the extremely energy-intensive and carbon-intensive production of cement and steel. In the operation of buildings, the main sources of emissions are energy consumption for heating, cooling, lighting and other needs related to the daily operation of buildings to increase environmental awareness and regulations. These include energy efficient technologies, low-carbon building materials, and design [1]–[10] and modelling [6], [11] - [17] according to sustainable principles. One of the most widely used and still developing branches of the construction industry is the production of chemical admixtures for cementitious composites. Chemical admixtures for concrete are special additives that are used to improve various properties of concrete mix. Plasticizers and superplasticizers increase the fluidity of the mix, allowing the amount of water to be reduced and increasing the strength of the concrete. Retarders delay cement setting, which is beneficial in situations where the concrete is transported over long distances or when working at high temperatures. Accelerators, on the other hand, speed up concrete hardening, which is important in winter conditions or for fast-paced construction projects. Aerating agents introduce microscopic air bubbles, increasing the frost resistance of concrete. Sealing admixtures improve the watertightness of concrete. Some of these substances, especially those based on polymers, may show resistance to biodegradation processes, implying a potential cumulative threat to ecosystems. Although they are rarely toxic to humans per se, their presence in water can have a detrimental effect on aquatic organisms [18]. Furthermore, the presence of these compounds in soil and groundwater can lead to environmental contamination. Therefore, it is essential to use these additives prudently, following environmental principles, to minimize their potential impact on aquatic and natural ecosystems. In recent decades, there has been growing interest in the use of natural organic compounds to produce plasticizers, as evidenced by the abundance of literature on the subject. [19]-[21]. The use of natural admixtures in the production of concrete confers a multitude of benefits, both in terms of technical efficacy and environmental impact. Primarily, these admixtures are capable of markedly enhancing the mechanical properties of concrete, in conjunction with nanoparticles [22]-[26], thereby conferring greater resistance to the detrimental effects of microorganisms. Furthermore, natural admixtures enhance the rheological properties and workability of concrete. Improved workability is associated with greater ease of pouring, pumping and forming concrete, as well as a reduction in the quantity of water required to achieve the desired consistency of the concrete mix. This, in turn, leads to an increase in strength parameters and a more homogeneous concrete structure. One of such substances is starch [20], [21], [27].

Starch is a polymer composed of glucose molecules and plays an important role in the plant and animal kingdoms as a major source of energy. Its structure is based on long polymer chains made up of glucose residues linked together by glycosidic bonds. It is found in the form of grains in various parts of plants, such as potato tubers, cassava roots or cereal seeds, including wheat, maize, and rice [20].

The process of extracting starch from raw plant materials involves the crushing of the raw material and subsequent separation of the starch from other plant using mechanical and chemical extraction methods. Once starch has been obtained, it can be subjected to various modification processes that alter its physicochemical properties [28]–[31]. In the construction industry, modified starch is used as an additive to modify the properties of building materials, particularly concrete. Starch modifications may include enzymatic breakdown into lower-molecular-weight components or chemical modifications, resulting in starches with different properties. Examples of applications of modified starch in the construction industry include improving the rheological properties of concrete, which can affect its workability and uniformity [18], [19]. In addition, starch can be used to improve the adhesion of paint coatings, plasters, or adhesive mortars for ceramic

tiles. Another potential application is the reduction of shrinkage in concrete, which is crucial to prevent cracking and deformation of structures. Additionally, modified starch can be used to enhance the water resistance of concrete or other building materials and increase their elasticity, which can be beneficial in structures susceptible to dynamic movements or vibrations. When combined with nanoparticles, it can provide a material that is resistant to microorganisms.

Modified starch is a crucial additive employed in the construction industry that can affect various technical and functional attributes of building materials, enhancing their performance and durability. Its versatility and modifiability make starch a valuable raw material in the construction industry, supporting sustainability and innovation in this field.

The objective of this research is to determine the impact of starch modified in the form of dextrin on the compressive strength and workability of cementitious composites.

2. Materials

2.1 Portland Cement

The study was carried out using Portland cement of grade CEM I 42.5R, obtained from Heidelberg Materials. Elemental analysis was performed using a Hitachi S-3400N scanning electron microscope equipped with an Ultra Dry EDS (Energy Dispersive X-Ray Spectrometry) analysis attachment (Thermo Scientific). The results of the tests are presented in Table 1.

Element	% Weight	% Atomic
С	15.84	26.69
0	36.64	46.36
Mg	0.52	0.43
Al	1.92	1.44
Si	10.84	7.81
S	0.48	0.30
K	0.54	0.28
Ca	32.50	16.41
Ti	0.10	0.04
Fe	0.62	0.23
Total	100.00	100.00

2.2 Admixtures used

The study was carried out using white dextrin, designated LU-1400-2 (d2). Dextrins are derivatives of native or modified starches. They are a completely natural and biodegradable substance, composed of derivatives of monosaccharides (glucose) linked by α -1,4-glycosidic bonds (Figure 1). They are derived from the hydrolysis of starch, which is the thermal treatment of potato flour with or without the addition of mineral acids or with the aid of enzymes.



Fig. 1. Structure of Dextrins

White dextrins exhibit a color similar to that of starch. They are formed by partial hydrolysis and drying of starch at 100°C. Hydrochloric or nitric(V) acids or a mixture of these are often used to acidify the compound. The physicochemical properties of these dextrins were determined based on internal studies, which are shown in Table 2. The moisture content of the commercial dextrins was determined using a MAC 50 IR weighing machine from Radwag. The viscosity of the dextrin solutions was measured using a HAAKETM ViscotesterTM 550 viscometer from Thermo ScientificTM.

The course of starch hydrolysis and the degree of saccharification of the final product can be controlled by determining the reducibility of the hydrolysate, which is defined as the content of reducing sugars DE (dextrose equivalent). The DE parameter describes the amount of reducing substances (as determined by Fehling's reagent) per gram of glucose contained in 100 grams of dry hydrolysate substance. The content of DE-reducing groups was determined using the Schoorl-Regenbogen method, following PN-78/A-74701.

The course of starch hydrolysis and the degree of saccharification of the final product can be monitored by determining the reducibility of the hydrolysate, i.e. the content of DE (Dextrose Equivalent) reducing sugars. The DE parameter describes the amount of reducing substances (as determined by Fehling's reagents) converted to glucose in grams per 100 grams of dry hydrolysate substance.

Tab. 2. Physicochemical parameters of dextrins.

Name	Symbol	Type	Moisture content [%]	DE	pH value of a 1% solution	Solubility at 20°C, %	Viscosity [Pa·s]
LU-1400-2	d2	white	9.4	4.25	3.6	< 65	0.21

3. Methods

The tests were carried out on mortars with doses of starch hydrolysates ranging from 0.25% to 0.40%, calculated in relation to cement weight. Standard quartz sand with a particle size of 0.125 to 2.00 mm, following the European standard EN 196-1, was used as an aggregate. The aggregate to cement was 1:3, and the water-cement ratio was 0.50, respectively, for all plasticizers tested. Admixtures were dissolved in mixing water. The amount of ingredients used, calculated per excipient, is presented in Table 3.

Tab. 3. Composition of cement mortar.							
w/c	Water[g]	Cement [g]	Aggregate [g]	Sum [g]			
0.50	225.0	450.0	1350.0	$\Sigma = 2025.0$			

The cement mixes were prepared using an automatic cement mixer according to the specifications outlined in EN 196-1. The consistency of fresh cement mortar was designated based on the guidelines outlined in EN 1015-3, using a flow table for mortars. This method involved measuring the diameters of the flow of cement mortars subjected to dynamic shocks.

To assess the strength of cement mortars following EN 196-1, several beams with a cross-sectional dimension of 40 x 40 x 160 mm were constructed. After 24 hours of mixing, the samples were disassembled and placed in a chamber containing water at a temperature of $20 \pm 1^{\circ}$ C. The compressive strength of the samples was then measured after 28 days of maintenance using a Walterbai test machine.

Viscosity measurements were performed using the Thermo ScientificTM HAAKETM ViscotesterTM 550, equipped with an MV-DIN rotor. The viscosity of 40% dextrin solutions was measured at room temperature with a rotational speed of 100 rpm ± 1 . Rheological tests on cement slurries were carried out for samples containing 0.5% of a modifying agent in relation to cement weight, as well as for the w/c ratio equal to 0.40 and 0.50, respectively. The aqueous solution of dextrin was added to an adequate amount of cement, and the mixture was stirred for 10 minutes using a mechanical stirrer. The viscosity of the slurry was then measured by applying rotor speeds in the range of 10 to 300 rpm ± 1 . Based on the viscosity measurements, the values of the the rheological parameters of cement slurries were designated using the Bingham and Herschel-Bulkley models [32], [33].

4. Results

4.1 The flow diameters

Figure 3 illustrates the flow diameters of the cement mix doped with d2 dextrin and commercial plasticisers P1 and P2 in amounts of 0.25-0.40, expressed as a percentage by weight of cement.



Fig. 2. Flow diameters of mortar cement with admixture d2 and plasticizers P1 and P2.

The reference value for the diameter of the spread was 19 cm. The addition of starch increased the flow diameter of the cement mixture by approximately 32, 34, 37 and 40%, respectively. The admixture of plasticizer P1 resulted in an 8% increase in the flow diameter, while for P2, the maximum increase was 16%.

4.2 Compressive strength

To assess the impact of plasticisers on compressive strength, cement mortar beams were manufactured. The resulting strength data is presented in Figure 3.



Fig. 3. Compressive strength of cement mortar beams with dextrin admixtures d2.

A series of mortars were prepared, with the addition of dextrin d2 at a concentration of 0.25%, 0.30%, 0.35% and 0.40% relative to the weight of the cement. After 24 hours, the samples were removed from the molds. The reference value for the compression strength of the beams was 49.0 MPa. The addition of 0.35% dextrin d2 resulted in an increase in compressive strength to 52.0 MPa, representing a 6% improvement compared to the reference sample.

In the subsequent phase of the study, the impact of commercial admixtures on mortar strength was evaluated. Commercial formulations were incorporated at a rate of 0.35% by weight of cement. Unfortunately, the compressive strength decreased to a value of 42.9 MPa for plasticizer P1 and a value of 43.3 MPa for plasticiser P2 (a decrease of 12.4% and 11.6%, respectively).

4.3 Rheology

Figure 4 presents the results of plastic viscosity tests conducted following the Bingham and Hershel Bulkley models, with w/c values of 0.50 (Figure 4.a) and 0.40 (Figure 4.b), respectively. For the sake of clarity, the values calculated according to the Bingham model will be provided directly in the test, while those according to the Hershel Bulkley model will be presented in brackets.



Fig. 4. Plastic viscosity of cement slurries for w/c = 0.50 (a) and w/c = 0.40 (b).

Figure 4.a, which shows the effect of d2 dextrins on the plastic viscosity of the cement slurry, reveals a notable reduction in viscosity compared to a reference value of 0.115 Pa·s (0.150 Pa·s). The addition of d2 dextrins to the cement slurry results in a viscosity value of 0.048 Pa·s and 0.062 Pa·s, respectively.

Furthermore, Figure 4.b for w/c = 0.40 reveals that the addition of dextrin d2 to the slurry results in a reduction in plastic viscosity, compared to a reference value of 0.177 Pa \cdot s (0.230 Pa \cdot s) to a value of 0.135 Pa \cdot s and (0.175 Pa \cdot s).

Figure 5 presents the results of the melt flow limit tests calculated according to the Bingham and Hershel Bulkley model for values of w/c = 0.50 (Figure 5.a) and 0.40 (Figure 5.b).



Fig. 5. Yield point of cement slurries for w/c = 0.50 (a) and w/c = 0.40 (b).

By analyzing Fig. 5.a for w/c = 0.5, it can be seen that the admixture of d2 dextrin to the cement slurry causes a significant reduction in the flow limit, compared to a reference value of 11.749 Pa-(11.279 Pa) to a value of 2.659 Pa (2.446 Pa).

A reduction in the w/c ratio to 0.40 (Figure 5.b) also reveals a decrease in the flow limit, compared to a reference value of $\tau_0 = 17.926$ Pa (17.209 Pa), to a value of 4.614 Pa (4.060 Pa).

The presented results of the flow limits and plastic viscosities calculated according to the Bingham and Herschel-Bulkley models are found to be different. As the shear rate increases, the viscosity decreases significantly (shear-thinning slurry). The increase in stress due to an increase in the shear rate causes the agglomerates or floccules to break apart and release the liquid inside, leading to liquefaction of the slurry. This material behavior is most accurately described by the Herschel–Bulkley model.

5. Conclusion

Incorporation of LU-1400-2 dextrin into cement mixtures yields several significant improvements in the properties of the material. These include an increased compressive strength and increased liquefaction.

Enhanced compressive strength: The addition of LU-1400-2 dextrin results in increased compressive strength of the cement mixtures. This enhancement suggests that dextrin may contribute to better particle bonding or may be beneficial in altering the hydration process.

Increased liquefaction: Incorporation of dextrin into fresh cement mixtures results in a notable increase in liquefaction. This enhanced liquefaction can facilitate improved workability of the cement mixture, making it easier to mix, pour, and shape. This is particularly beneficial in construction processes that require precise application and smooth finishes.

Furthermore, the incorporation of dextrin into the cement slurry significantly reduces both the flow limit and the plastic viscosity. This reduction in resistance to deformation and flow facilitates easier pumping and spreading of cement. The lower plastic viscosity also indicates that less energy is required to maintain the flow, which potentially leads to more efficient processing and application.

In summary, the use of LU-1400-2 dextrin in cement mixtures improves mechanical properties like compressive strength, and improves rheological properties such as liquefaction, flow limit, and viscosity. These modifications can facilitate the more efficient and versatile use of cement in a variety of construction applications, potentially reducing costs and improving the quality of the final product.

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