



Research article

Cold-curing mixtures based on biopolymer lignin complex for casting production in single and small-series conditions

Falah Mustafa Al-Saraireh*

Department of Mechanical Engineering, Faculty of Engineering, Mutah University, Jordan

* **Correspondence:** Email: f_saraireh@mutah.edu.jo.

Abstract: This study proves that lignin-based biopolymer materials can be employed as starting materials for the synthesis of novel casting binders that fulfill the current level of characteristics. The optimal concentration of the binder in the mixture was experimentally determined to be 5.8%–6.2%. It has been demonstrated in practice that the employment of ammonium salts as a technical lignosulfonate (TLS) modifier can result in the provision of cold (room temperature) curing of a mixture based on them. It was proposed to use as a technological additive that boosts the strength characteristics of a mixture of substances carboxymethyl cellulose (CMC). In a variety of adhesive materials, it is utilized as an active polymer base. The concentration limits for using CMC in the mixture are set at 0.15%–0.25%. To improve the moldability of the combination, it was suggested that kaolin clay be used as a plasticizing addition. The concentration limits for using a plasticizing additive are set at 3.5%–4.0%. The produced mixture was compared to the analog of the alpha-set method in a comparative analysis. It was discovered that the proposed composition is less expensive, more environmentally friendly, and enables the production of high-quality castings. In terms of physical, mechanical, and technological properties, the created composition of the cold curing mixture is not inferior to analogs from the alpha-set method. For the first time, a biopolymer-based binder system containing technical lignosulfonate with the addition of ammonium sulfate and carboxymethyl cellulose was used in the production of cast iron castings on the case of a cylinder casting weighing 18.3 kg from gray cast iron grade SCh20. Thus, it has been proved possible for the first time to replace phenol-based resin binders with products based on natural polymer combinations. For the first time, a cold-hardening mixture based on technological lignosulfonates has been developed without using hardeners made of very hazardous and cancer-causing hexavalent chromium compounds. But is achieved through a combination of specialized additives, including kaolin clay to ensure the mixture can be manufactured,

ammonium sulfate to ensure the mixture cures, and carboxymethyl cellulose to enhance the strength properties of the binder composition. The study's importance stems from the substitution of biopolymer natural materials for costly and environmentally harmful binders based on phenolic resins. This development's execution serves as an illustration of how green technology can be used in the foundry sector. Reducing the amount of resin used in foundry manufacturing and substituting it with biopolymer binders based on technological lignosulfonates results in lower product costs as well as the preservation of the environment. Using lignin products judiciously can reduce environmental harm by using technical lignosulfonates, or compounds based on technical lignin. The combination is concentrated on businesses with single and small-scale manufacturing because it is presumable that this is merely the beginning of the investigation. This study confirms the viability of creating a cold-hardening combination based on technical lignosulfonates in practical applications and supports this with the castings produced, using the creation of a gray cast iron cylinder casting as an example.

Keywords: foundry; binding materials; binding capacity; technical lignin; formability; crumbling; carboxymethyl cellulose; kaolin; ammonium sulfate; casting

1. Introduction

The processes used for the manufacture of castings are the foundation of modern mechanical engineering. 60%–90% of the structure of everything that travels, flies, or floats contains parts made by one or more casting methods [1].

Modern foundry technologies are manufacturing processes that depend on using materials unique to foundry production and are processed on highly specialized equipment to produce a high-quality casting of any configuration and size with a predetermined set of properties [2,3].

It has been established that the factor determining the name of a specific foundry technique is a critical component that dictates the technological feasibility of casting manufacturing [4]. For example, they may say “manufactured by the amine process”, “manufactured by the alpha-set process” or “manufactured by the sand-clay mixture (SCM) process”, with the type of casting binder employed deciding the name of the technique in each case. This shows that foundry binder material has the most technological significance [5].

The main production of castings is concentrated in a relatively small number of enterprises, accounting for approximately 25%–30% of the total list [6], and is characterized by a mass type of production, with all technological operations performed on specialized technological complexes, such as automatic or automated conveyor lines and special machines (for example, Laempe core machines for the production of foundry cores) [7].

Depending on the engineering industry, they account for 65%–95% of casting production. However, 70%–75% are small and medium-sized businesses specializing in repair or casting manufacturing [8]. Despite the fact that the specifics of single and small-scale manufacturing differ fundamentally from those of mass production, the market demands the same expensive technology, particularly binding materials, as that of large companies with mass production. In most cases, these are phenolic-based resins, which are both prohibitively expensive and hazardous to the environment. What is inherent in mass manufacturing is not necessarily successful in the production of a single one.

Taking into consideration price increases, the cost of a kilogram of resin on a phenolic foundation ranges from 7–10 euros, depending on the brand and use [9]. This style of work is becoming increasingly popular for sociopolitical reasons [10].

It is recommended to consider alternative solutions that could overcome this disparity, and given the difficulties associated with embedding, introducing new materials into the technological chains of large enterprises of the mass production type, try to apply innovations in economically flexible conditions. Castings on a single and tiny scale [11,12].

To provide a low-cost, environmentally friendly, and efficient casting binder for use in industrial firms with small-scale and single-piece casting production.

The development should be simple to use and effective, in terms of the results obtained. Initially, it was decided to exclude the complex process of preparing a binder during production, which could require the creation of an extra technological module at the enterprise with associated costs and regulations. This decision was made to ensure efficiency and avoid unnecessary expenses. It is expected that the suggested new binder material's matching "place of assembly" would be the location for preparing the casting mix itself, that is, a technological unit, a mixer (a blade, roller, etc.).

It has been proposed that a natural biopolymer complex based on technical lignin might be employed as a starting point for the synthesis of novel binders. This material is available on the market for secondary substances of techno-genic origin in numerous modifications of technical lignosulfonates (TLS)-liquid, powder, solid, in the physical condition, and in various chemical compositions. This limits the variety of options for developing fundamentally new products.

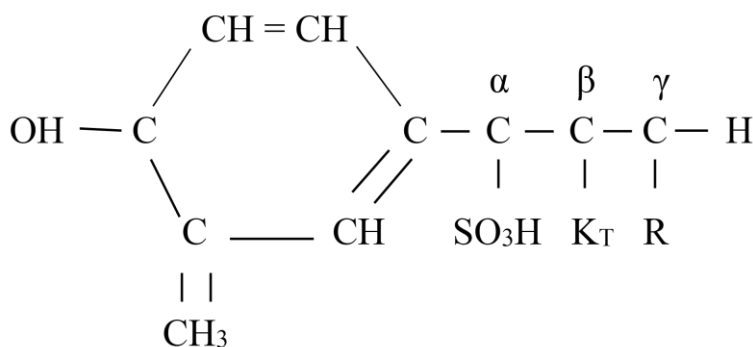
Technical lignin, in its different forms, is a large-scale waste product of vegetable raw material processing. The pulp and paper sector is one of the main producers of this substance. Sulfate, sulfite, and hydrolytic lignin are separated according to the process of wood delignification.

The total volume of this substance produced in the globe is believed to be up to 100 million tons, with the majority of it discharged as wastewater and contributing significantly to the degradation of the world's water basin [13]. There is currently no rational integrated method for dealing with the problem of technical lignin usage. This defines the prospects of this area of study, the resource base required for product development, the low cost of raw materials, and the likelihood of future price increases. Lignin acts in plant tissue by connecting bonding cellulose fibers and ensuring the consequent strength. As a result, this substance is predisposed genetically to be employed as a binder [14].

The formula of lignin $(C_{31}H_{34}O_{11})_n$ given in the literature [15] is approximate since it depends on the specific type of plant tissue, which introduces uncertainty into the properties of materials and products based on it.

Technical lignosulfonates are a product of plant tissue delignification and significantly depend on this process, which predetermines the instability of their quality characteristics. In essence, this is due to the reluctance of casting manufacturers to use this material [16].

In general, the structural formula of lignosulfonate is shown in Figure 1.



Where:

R: -OH, =NH, -COOH, etc.;

K_T: cation of the cooking base Na Ca, Ca = Na, Mg, or ammonium.

Figure 1. The structural formula of liginosulfonate.

Lignosulfonates are the common name for salts of liginosulphonic acids. They are natural water-soluble sulfonic derivatives of lignin [17]. It should be noted that the exact structure of liginosulfonates has not been established to date, which sufficiently explains the increased interest in studying the substance in laboratory conditions [18].

Creating, and utilizing highly advanced, reasonably priced, and environmentally friendly binder materials is pertinent to the foundry sector.

The technologies utilizing cold-hardening mixtures and the so-called chemical-technological systems (CTS) process, one of which is the alpha set process [19], are the most hopeful ones for the production of castings.

Based on the ability of the sand mixture to cure without the use of heat and under the influence of a catalyst, the cold-hardening sand-casting method was developed. Nevertheless, this method has a number of benefits, the overview of which is shown in Table 1.

The listed advantages of CTS processes dictate the need for a more detailed study of the processes of structure formation of mixtures with TLS for the practical implementation of these advantages on mixtures with their use.

Table 1. Summary of the benefits of CTS methods for casting production in foundries.

No.	Index	Content
1	Ensuring the surface quality of castings	The cleanliness of the surface is ensured, the accuracy of the geometric dimensions of the casting is observed and the high quality of products is achieved.
2	Ensuring the dimensional accuracy of castings	There is no deformation during the production of molds and the number of defects is minimal.
3	Cost reduction	Due to the use of simpler equipment, the cost of production is reduced.
4	Possibility of automation	The simplicity of technological operations provides the possibility of automating the process of preparing mixtures.
5	Reducing energy costs	Due to the absence of the operation of heating the mold or core.
6	Reduction of labor intensity	Reduces the complexity of finishing operations.

2. Materials and methods

Let us consider the possibility of using TLS as an initial binder material in CTS-casting production processes. Figure 2 shows the structural and logical scheme of the research.

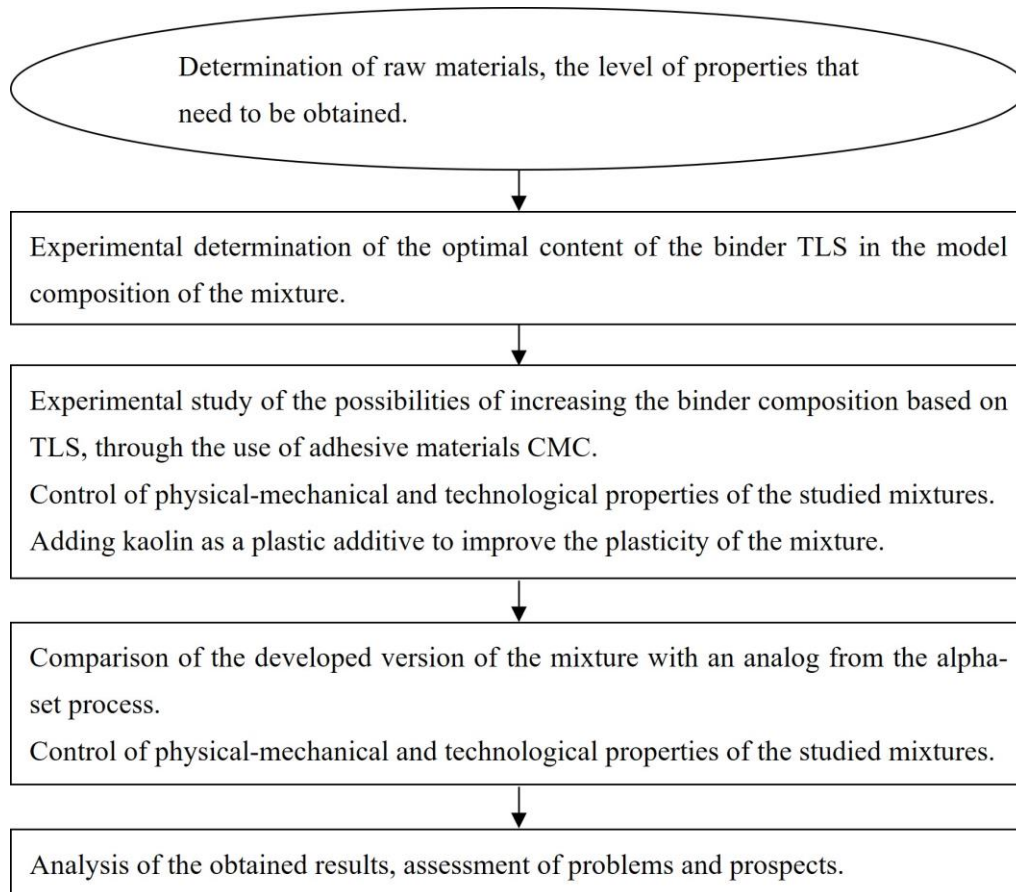


Figure 2. Structural and logical scheme of the research.

As the primary binding agent for the study, liquid technical lignosulfonate made utilizing the sulfite pulping technique was used. Ammonium sulfate, CMC, and kaolin clay are some of the technological additives used.

For research experiments, we used carboxymethylcellulose (CMG, croscarmellose, cellulose glycolic acid, $[C_6H_7O_2(OH)_{3-x}(OCH_2COOH)_x]_n$, where $x = 0.08-1.5$) is a cellulose derivative in which the carboxymethyl group ($-CH_2-COOH$) combines hydroxyl groups of glucose monomers [20,21].

The strength characteristics were measured using a POLITEST MP-01 mechanical testing machine.

The preparation of mixtures and control of their properties was carried out according to standard methods adopted in the foundry industry:

1. Mixtures for testing were prepared by mixing in batch mixers brand 018M.

2. Tensile strength tests were conducted on dried samples in the shape of an “eight” that were prepared in core boxes of model 037M. To do this, the prepared mixture is poured into the core box of the specified model, a bulk frame is installed, the mixture prepared in accordance with the provided recipe is poured, a tamping head weighing 750 g (± 10 g) is placed on it and the box is then placed on a

laboratory pile driver and stuff by a triple impact of 63.5 g weight, the mixture has been compressed, which falls on a pile driver from a height of 50 mm. After compaction, the “figure eight” sample is removed from the core box and cured in an oven at a temperature of 180 °C for an hour, after which the samples are cooled at room temperature and tensile tested on a POLITEST MP-01 mechanical testing machine [22,23].

3. Determination of compressive strength for cylindrical samples (preparation in a cylindrical sleeve, stuffing on a laboratory impact tester, extraction) was carried out in a similar way, according to a standard foundry method. The resulting cylindrical samples prepared from the compositions of cold-hardening mixtures were removed from the sleeve (core box), cured under room conditions in the laboratory, and subjected to control (determination of compressive strength) on the POLITEST MP-01 mechanical test machine [24].

4. The mixture’s survivability was determined by its exposure time from the moment the prepared mixture was released from the mixer, until the moment the sample was made, its depravity was reduced by 30% compared to its maximum indicators.

5. The crumbling (friability) was controlled according to the standard method (23409.9-78) the prepared cylindrical sample was weighed and then placed in the central part of the drum with a diameter of 110 mm in a horizontal plane with a rotation speed of 60 ± 5 rpm, which is rotated when testing raw samples within 30 s. Then, the drum was stopped and the sample was removed and weighed again. The test was carried out using three samples [25,26].

The final crumbling (X) as a percentage is calculated by the Eq 1:

$$X = (m - m_1)/m \times 100\% \quad (1)$$

where m is the mass of the sample before testing, g;

m_1 is the mass of the sample after testing, g.

6. Formability characterizes the ability of the sand to reproduce the configuration of the model. The accuracy of the resulting casting depends on this indicator. Formability characterizes the ability to fill a variety of pockets and hard-to-reach places with free backfill. As a rule, the concept of “flowability” is synonymous in meaning with formability. Formability is determined by sifting the mixture in a mesh drum with a diameter of 100–110 mm with a mesh cell size of 2.5 mm, drum rotation speed of 60 rpm, sample weight of 200 g, and rotation time of 10 s. The formability is estimated by the formability factor F_k (%), according to the Eq 2:

$$F_k = (m_2/m_1) \times 100\% \quad (2)$$

where m_2 is the mass of the mixture passed through the grid;

m_1 is the initial mass of the sample.

3. Results and discussion

The kinetics of strength development was examined during the liquid technical lignosulfonate-based cold curing method. They were initially changed based on the existing, widely accepted theories concerning the chemistry of the interaction of TLSs with ammonium salts [27]. The modifier ammonium sulfate was utilized. The fundamental cause of this chemical reaction is that divalent ammonium creates cross-links in the oligomeric chains of TLS as a result of the substitution of sulfur

groups, which results in the formation of a specific spatial frame polymer structure and the cold curing of the sand mixture.

The ideal amount of ammonium sulfate to have in the combination is 2%, according to preliminary investigations [28,29]. Initially, a binder material was made by adding ammonium to the TLS and vigorously combining the mixture at room temperature (20–25 °C). The binder is ready for use after being held for 30–40 min.

The ideal level of modified TLS in the mixture was first established. The trials were conducted on model mixtures that contained, in turn, 4%, 6%, 8%, and 10% of modified TLS. This was done to investigate how strength develops kinetically. Figure 3 shows the experiment's results.

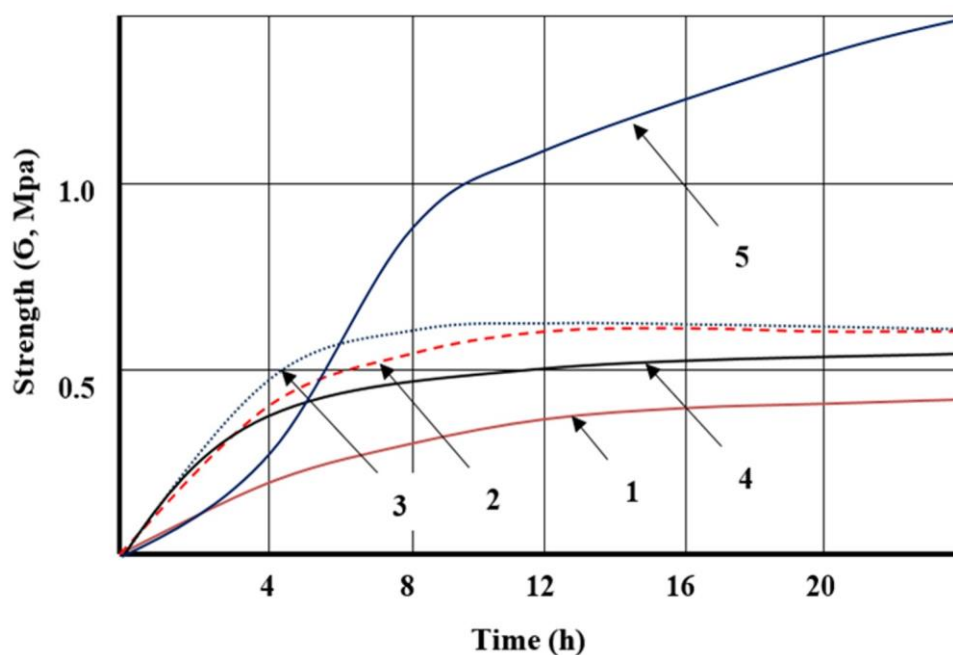


Figure 3. Strength of solidification of mixtures with TLS-modified ammonium sulfate, with different content of the binder in the mixture. The content of the binder in the mixture, respectively: line 1 contains 4% TLS in the mixture, line 2 contains 6% TLS in the mixture, line 3 contains 8% TLS in the mixture, line 4 contains 10% TLS in the mixture, line 5 mixture according to the alpha set process.

According to the results of all the tests, 6% of TLS modified with ammonium sulfate is the ideal concentration and the strength indicators were attained after 24 h. There is a 0.62 MPa exposure. It is possible that this outcome will not always be enough to secure great casting. It was proposed to add CMC, which is frequently used as an active adhesive basis in various goods, to the composition of the cold-hardening mixture with TLS modified with ammonium sulfate in order to boost the strength of the mixture.

It was discovered through a series of search tests that CMC, in the form of a powder, can be added directly to the filler, i.e., quartz sand, during the creation of the mixture. According to calculations, the combination should contain between 0.05% and 0.35% of CMC in order to achieve maximum strength and experience. 0.1%, 0.2% and 0.3% CMC in the mixture was the subject of the investigation. The

research aimed to select the ideal CMC value for the mixture's composition. Figure 4 displays the overall findings from the experiments.

It has been established that adding CMC to the casting mixture greatly raises the strongest indicators of the model binder compositions.

The physical-mechanical and technical characteristics of the studied mixtures were observed over the course of experimental studies. Summary information on the composition of mixtures and their physical, mechanical, and technological properties is shown in Table 2 based on the outcomes of the application of the designated research algorithm [30].

Evaluating the results obtained, we can say that composition 6 (see Table 2), in terms of a set of properties, is closest to the option that could be applied in the practice of casting production and, in the future, recommended for the production of a certain casting segment.

The disadvantage of this composition of the mixture is the need to improve formability. This indicator determines the dimensional accuracy and reproduction of the configuration of the future casting and therefore should be as close to the ideal as possible.

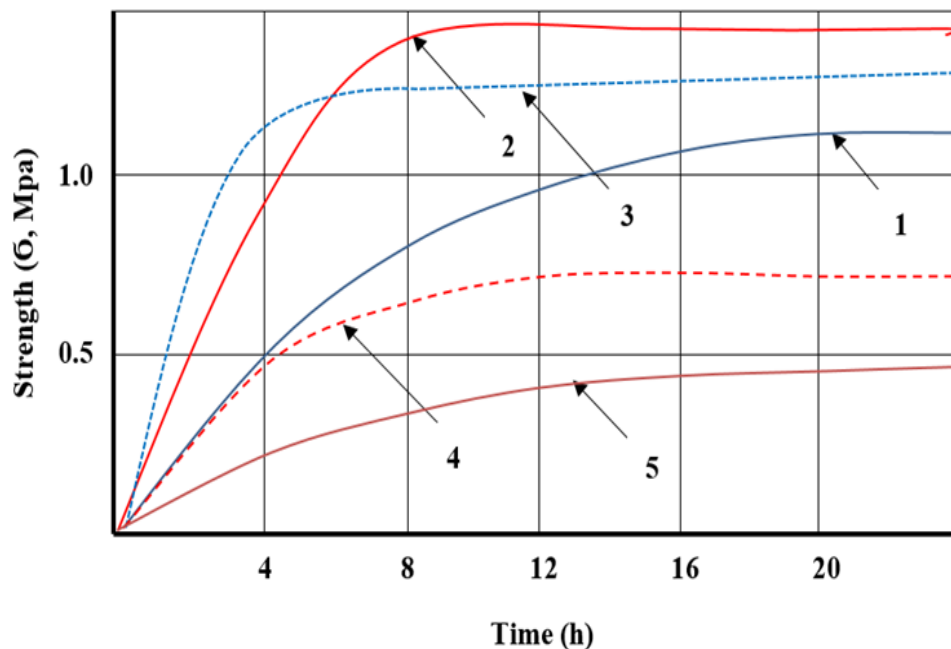


Figure 4. The strength of mixtures increases when CMC is added, with the ideal amount of lignosulfonate binder (6% TLS, 0.2% ammonium sulfate) in the mixture. The amount of binder in the combination is as follows: line 1 contains 0.1% CMC, line 2 contains 0.2% CMC, line 3 contains 0.3% CMC, line 4 contains optimal binder content without CMC, and line 5 contains minimum binder content without CMC.

Table 2. Component compositions and indicators of physicochemical and technological properties of the developed mixture compositions.

No.	Composition of the mixture, wt %			Physical and mechanical properties		Technological properties		
	TLS with 2% ammonium sulfate added	CMC	Sand	Compressive strength after 24 h, MPa	Tensile strength of dry samples, MPa	Vitality, min	Peeling, %	Formability, %
1	4		96	0.42	0.57	11	1.39	72.9
2	6		94	0.61	0.72	13	0.88	89.1
3	8		92	0.61	0.67	15	0.93	90.1
4	10		90	0.56	0.47	14	1.06	92.8
5	6	0.1	94	1.27	1.82	11	0.18	93.1
6	6	0.2	94	1.50	2.02	11	0.11	94.9
7	6	0.3	94	1.33	2.00	10	0.10	98.0

It was suggested that molding kaolin clay be added to the mixture's composition in order to increase formability. It has been proven through experimentation that 3.7% clay is the ideal amount to have in the combination. This amount was determined experimentally, corresponding to the maximum formability index, with the maximum strength characteristics of the mixture, and at the same time, with a minimum binder with the composition of the mixture.

According to the test results, the primary model mixture in this instance was mixture 6 (see Table 2). The most effective strength and technological property indicators were seen with a given amount of clay. The composition of the proposed combination based on TLS was compared to a mixture created utilizing the alpha-set process technology. The alpha-set process was chosen as the basis for comparison because the enterprise where the development was originally supposed to be tested uses exactly this technology. In the future, a wider experimental coverage is expected to compare the proposed approach with other polymer systems. In Table 3, the results of the experiments are presented.

In terms of technical indicators, the proposed mixture's composition was comparable to the base one currently in use, however, it stood out because it lacked components that would have caused highly toxic and carcinogenic substances to be produced during the technological process of making castings [31,32].

During pilot industrial testing of the proposed composition of the mixture (composition of mixture 6, see Table 2), casting "cylinder" was obtained, weighing 18.3 kg, from gray cast iron (Figure 5), used in the manufacture of compressor technology. In the production of castings for the process of testing, the proposed composition was used as molding sand.

The combination of kaolin clay with lignosulfonic acid in the composition of the mixture led to the effect of self-precipitation, i.e., spontaneous destruction of the mixture after pouring. After pouring cast iron, the mixture spontaneously spilled out of the intercostal cavities on the surface of the casting. This effect is apparently caused by increased thermal degradation of the lignosulfonate component in the composition of the mixture upon contact with molten metal in the area of the casting ribs so that the resulting gases destroyed/weakened the mold elements after the casting crystallized. At the same time, even the presence of clay did not lead to a deterioration in knockout and an increase in the knockout work.

Table 3. Comparative characteristics of the properties of the proposed composition of the mixture, with a mixture based on the alpha-set process.

Composition of the mixture, wt %	Physical and mechanical properties		Technological properties		
	Compressive strength after 24 h, MPa	Tensile strength of dry samples, MPa	Vitality, min	Peeling, N/mm %	Formability, %
According to the proposed option, the composition: 1. Quartz sand-94% 2. TLS (with 2% ammonium sulfate added)-5.5%, CMC-0.2%, kaolin clay-3.7%	1.53	2.11	15	0.13	98.9
According to the alpha set process, the composition: 1. Sand quartz-8% 2. Resin brand ALFABOND-2.0% 3. Ester hardener-25%, from the amount of resin	1.51	2.69	7	0.15	99.3



Figure 5. Casting “cylinder”, obtained from the proposed composition of the mixture.

From an economic point of view, the cost of a kilogram of German-made Alfa bond resin is 5.8–6.3 euros and the cost of a kilogram of TLS is 0.1–0.2 euros, which is not commensurately less than the cost of phenolic resin. Significant economic benefits will be maintained despite the fact that TLS is used in the mixture almost three times more than resins, the remaining ingredients are used in small quantities and they will not be able to significantly change the balance.

In contrast to resin mixtures created using the alpha-set procedure, the suggested mixture’s components are non-toxic, have a much longer shelf life, are simple to handle, fireproof and nonexplosive [33,34]. The cast iron castings produced while using the proposed mixture as a casting mold were easily removed from the mold after a light tap with a hammer caused the mixture to pour out of the cavities.

No offensive smells were noticed during the mixing, pouring of molten iron into a mold, cooling of the casting inside the form, or knocking out that followed. Casting surfaces were spotless and free of extraneous inclusions and burns, which can be seen in (see Figure 4).

A step-by-step strategy for the development of this approach is advisable, the volumes of lignosulfonate produced are such that this material, with a reasonable approach, can become an alternative to using a variety of phenolic-based resin binders in the future, both cheaper in cost and more environmentally friendly in use. At the initial stage, it was decided to focus the development on the area of single and small-scale production of castings as the simplest in practical implementation. In our opinion, the use of technologies using technical lignin in their technological cycle is the practical implementation of the concept of green technologies [35].

The novelty of the work lies in the fact that:

1. For the first time in the production of cast iron castings, on the example of a “cylinder” casting, weighing 18.3 kg. From gray cast iron grade SCh20, the alpha set process binder complex, using alpha bond resin and ester hardener, was replaced with a biopolymer-based binder system, including technical lignosulfonate with the addition of ammonium sulfate and carboxymethyl cellulose. Thus, the idea of replacing phenol-based resin binders with products based on natural polymer complexes has been realized for the first time.

2. For the first time, a cold-hardening mixture based on technical lignosulfonates has been created that does not use highly toxic and carcinogenic compounds of hexavalent chromium as hardeners but is implemented by a combination of special additives, in particular ammonium sulfate, to ensure the curing of the mixture, and carboxymethyl cellulose, which performs the function of enhancing the strength characteristics of the binder composition with the additive kaolin clay to ensure the workability of the mixture.

3. The formulation of cold-hardening mixtures based on technical lignosulfonates and carboxymethyl cellulose was experimentally determined, which regulates the quantitative characteristics of the binder complex. It is proposed to use 5.5% technical lignosulfonate, with the addition of 2% ammonium salts (ammonium sulfate) in combination with 0.2% carboxymethylcellulose and the addition of kaolin clay in the amount of 3.7%.

4. An algorithm for the use of cold-hardening mixtures based on technical lignosulfonates and carboxymethyl cellulose has been developed as the basis for further development and promotion of this direction. In particular, the procedure for using the proposed components in the process of the technological cycle has been established, using the example of the manufacture of the “cylinder” casting.

The significance of the study lies in the fact that:

Expensive and environmentally hazardous binders based on phenolic resins have been replaced with biopolymer natural materials. The implementation of this development is an example of the practical use of green technologies in the foundry industry. Reducing the use of resin binders in foundry production, and replacing them with biopolymer binders based on technical lignosulfonates, is not only a reduction in product costs but also the preservation of the health of enterprise personnel from the constant action of highly toxic emissions, as well as a significant reduction in the carbon footprint formed during the production of resins phenol-based. And vice versa, the use of technical lignosulfonates or materials based on technical lignin is the prevention of damage to the natural environment through the rational use of lignin products.

Recommendations for the future:

It is assumed that this is only the initial stage of research. For this reason, the mixture is focused on enterprises with single and small-scale production. This study confirms the possibility of successful practical application of the formulation of a cold-hardening mixture based on technical lignosulfonates, and confirms this with the obtained castings, using the example of making a cylinder casting from gray cast iron.

4. Conclusions

- It has been proven that lignin-based biopolymer materials can be employed as a starting material for the synthesis of novel casting binders that fulfill the existing level of characteristics when casting molds are created from the suggested mixture. Casts made of cast iron were simple to remove.
- Experimentally, a binder concentration of 5.8%–6.2% was found to be the optimum quantity in the mixture.
- It has been proven in practice that the use of ammonium salts as a TLS modifier can lead to the provision of cold (at room temperature) curing of a mixture based on them.
- It is proposed to use carboxymethyl cellulose, which is used as an active polymer base in a variety of adhesive materials, as a technological additive that increases the strength characteristics of a mixture of substances.
- The concentration limits for the use of CMC in the mixture were determined at the level of 0.15%–0.25%.
 - It is proposed to use kaolin clay as a plasticizing additive to increase the moldability of the mixture.
 - The concentration limits for using a plasticizing additive were determined at the 3.5%–4.0% level.
 - A comparative analysis of the developed mixture was carried out in comparison with the analog of the alpha-set process. It was found that the proposed composition is cheaper in cost, more environmentally friendly, and allows you to produce high-quality castings.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

The author declares no conflict of interest.

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