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Research of the New Laboratory Methodology for Measuring the Disintegration of Mixtures with Inorganic Binders Hardened by Dehydration

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Abstract

The paper deals with the possibilities of using alkali silicate based inorganic binders for automotive industry aluminium castings production. In recent years, inorganic binders are coming back to the foreground and their manufacturers are developing new processes, which are starting to progressively supersede organic binder systems. Paper describes known knowledge about classic alkali silicate binders with focus on hardening processes and on improving their technological properties. Trends from the area of development and the use new alkali silicate based inorganic binders are also shortly described. As part of the experimental work, specific methods of producing samples were developed, with the help of which properties such as disintegration were subsequently evaluated by measuring abrasion and residual strengths. Characteristics such as residual compressive strength or shear strength at different thermal loads were also evaluated. When comparing the laboratory results with the results of de-coring in real conditions, a high degree of correlation was achieved, which makes it possible to determine the optimal recipe/procedure for the production of geometrically complex cores.

Keywords: Inorganic binder, Alkali silicate, Hot curing process, Automotive industry

1. Introduction

1.1. Technological procedures to produce cores for large-scale production of castings for the automotive industry

This paper deals with production of high-quality cylinder head castings. For the production of cylinder head castings, several types of sand cores are used, which can be produced by different technologies [1-4]. These are the cores of the intake and exhaust channels, water space cores and oil space cores, which are part of the cover cores with the pouring part (Fig. 1).

The cores are produced using the technology of blowing a sand mixture into the core boxes. Depending on the type of technology used, the design of the blowing machine and the core box also differs. For the production of cores using Hot-Box or Warm-Box technologies, it is necessary to have heated core boxes – most often heated by gas burners, less frequently by electric heating. On the other hand, in the production of cores using the Cold-Box technology, it is necessary to have a gasification plate and a device for the gasification of the catalyst – amine.

1.2. Alkaline silicates

The use of solutions of alkaline silicates, mainly sodium and partly potassium silicates, known as sodium or potassium water glasses, in the foundry industry was presented in the patent of Dr. L. Petržela in 1947.

Aqueous sodium silicate, generally described by the chemical formula $xNa_2O.ySiO_2$, is among the most widely used and popular inorganic binders in the foundry industry for the production of environmentally friendly molds and cores [5-10]. Foundry molding mixtures prepared using alkaline silicates are relatively inexpensive compared to molding compounds with organic binders and have several other advantages, but they are also characterized by their undesirable properties such as high sensitivity to air humidity or high residual strength after casting, which is associated with impaired disintegration of molds and cores, and also the regeneration of mixtures with water glass is problematic [11-13]. Moreover, the $Na_2O.SiO_2$ system tends to react with the silica opening material and the result is the formation of a sintered molding mixture when in contact with hot metal. These properties are particularly undesirable when casting thin-walled castings from low-melting alloys and compared to organic binders, they represent a major disadvantage when using inorganic binders [14-17].

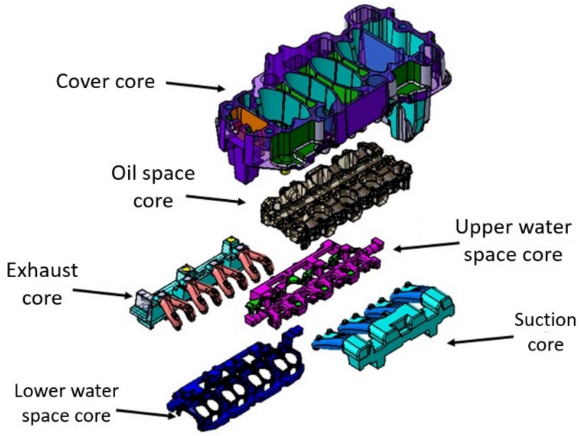

Fig. 1. Cores for the production of cylinder head castings

Together with the ever-increasing requirements for environmental protection, there is a need to use environmentally acceptable inorganic binders based on alkaline silicates, the technological properties of which are partially improved by chemical modification, while their impact on the environment remains minimal [18,19]. The formation of sinterings due to the interaction between the $Na_2O.SiO_2$ system and silica opening material can be reduced by lowering the binder content, but under the assumption of improved binder properties [19,20]. The past decade has been characterized by a renaissance of interest, especially by foreign foundries, in the use of inorganic binder systems. Initially, it was due to the already emphasized reason of increasing demands for environmental protection, but recently it is also due to the demands of car and engine designers in terms of improving their performance. These requirements are gradually met by the successful development and implementation of new processes based on modified alkaline silicates for the manufacture of cores to produce high-quality castings for the automotive industry [21].

1.3. New laboratory methodology for measuring the disintegration of mixtures with inorganic binders hardened by dehydration

After casting and de-coring, foundries have a major problem with un-cored channels in the lower water space of the castings, which results in high amounts of rejected products, as the channels cannot be additionally cleaned of the un-cored mixture in any way that would suit large-scale conditions. The occurrence of this problem indicates a large reserve in the setting of the formula from the point of view of the mixture disintegrability. The image of un-cored channels in the casting after cutting (a) and a detail of the residual mixture in the water space (b) are shown in Fig. 2.

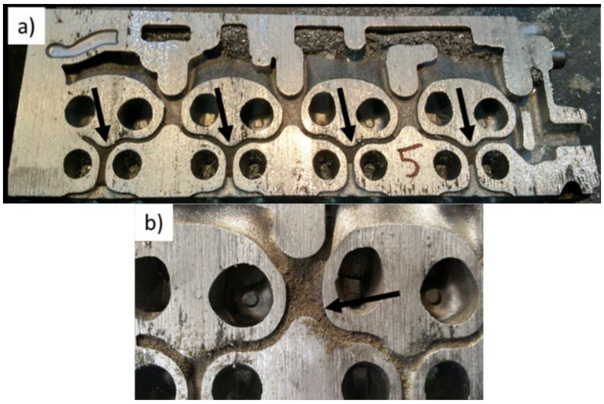

Fig. 2. Un-cored casting after cutting through the plane of the: (a) Water space; (b) Detail

The aim of this work is to propose a reliable and simple laboratory method for assessing the disintegrability of mixtures with inorganic binders hardened by the dehydration process. The result of the proposed methodology will be a clear criterion for the mixture disintegrability after casting, according to which it will be possible to design a formula that will guarantee the complete de-coring of the channels of the casting water space. The methods used so far to measure the disintegrability of the mixture are designed mainly for chemically hardened mixtures (CO_2 process, ST mixtures) and for binder systems used for iron alloys, for which these mixtures were more commonly used. The methods use either standardized rollers to measure the compressive strength after a given thermal load or consist of casting these rollers as cores placed in molds made of the same mixture [22-23]. For these reasons, the currently available methods are not suitable for assessing the disintegrability of new inorganic binder systems used for casting aluminum alloys into metal molds, and therefore it was necessary to create a new methodology that will meet the conditions of use in foundries. The values of residual strength, shear strength and abrasion of the cores may be suitable indicators to quantify degree of mixture disintegrability of these new inorganic binder systems and they are the main focus of this paper.

2. Description of used methodologies and results

The method of measuring the residual strength was tested as the only one of the usual methods, because only rollers made of the tested mixture, a resistance furnace and a universal strength device are needed for the measurement itself. For the production of rollers, a device designed for the production of samples from the Hot-Box system by the Polish manufacturer Multiserw Morek was used, which does not have the option of blowing with hot air. Therefore, the hardening time for the production of rollers had to be determined experimentally. The core temperature was set to 180 °C, and the blowing time to 0.5 s. Fig. 3 shows samples and shell thickness after different hardening times. It can be seen that at times 25 and 50 seconds the shell was too thin, which would result in more difficult sampling and handling with the risk of deformation. 100 seconds proved to be the optimal hardening time. The outer shell was strong enough to handle the sample. Based on practical experience, a shell thickness of approximately 10 mm can be recommended. The rest of the unhardened mixture inside hardened during cooling of the sample under the influence of accumulated heat, as is also the case in the production of cores with thick cross-sections under practical conditions.

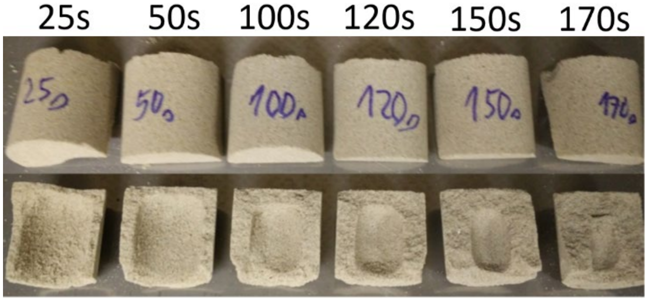

Fig. 3. Shell formation depending on curing time

The experimental determination of the optimal parameters to produce roller samples was followed by the measurement of disintegrability using the residual strengths. For this measurement, a mixture containing 2.0 % Inotec EP4158 and 0.9 % Promoter WJ4000 with SH32 opening material was chosen as a reference. In the available literature, the times required for the thermal load of the rollers at the given temperatures are not mentioned in the procedure for measuring the residual compressive strength. This is why these times were experimentally determined for temperatures of 200, 400, 600 and 800 °C. A thermocouple (type K) was inserted into the center of the roller, which was connected to the device for recording values. Subsequently, the furnace was heated to the required temperature, and after inserting the sample, the time it took for the sample to heat up in the center to the required temperature was monitored. Analogously, the times required for complete heating up of the rollers were determined for other temperatures, and the results are shown in Table 1.

Subsequently, the residual compressive strengths of the reference mixture at the given temperatures were measured. The resulting graph of residual strengths is shown in Fig. 4.

Table 1.
The times required to heat the roller to the given temperature.

Temperature [°C]	Temperature holding time [min]
200	26
400	30
600	27
800	53

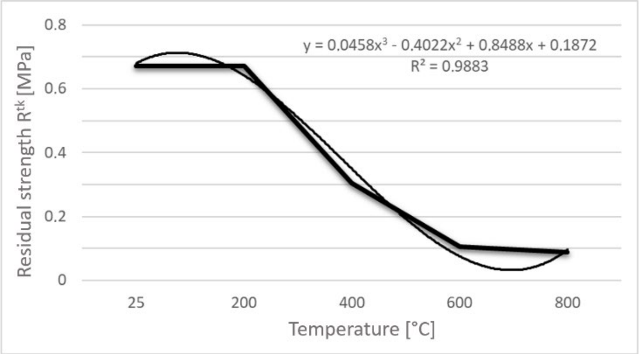

Fig. 4. Residual strengths for a mixture of 2.0 % Inotec EP4158 and 0.9 % Promoter WJ4000

From the graph in Fig. 4 it can be seen that the samples without thermal load had a high compressive strength above the measurable range of the device (0.13 – 0.67 MPa). Samples at a temperature of 200 °C, at which thermal degradation of the binder bridges did not occur but, on the contrary, their additional dehydration could occur, had a high residual strength, which could neither be measured by the strength device used. This confirms the high compressive strengths when using the dehydration hardening method compared to chemical reaction hardening. As the temperature increases further, a decrease in residual strengths is visible, while according to the regression equation, the minimum should be at 700 °C. This commonly used method was evaluated as unsuitable for assessing the disintegrability of mixtures due to the insufficient measurable range of the compressive strength of the rollers with the given device and, at the same time, due to the irregular breaking of the rollers during the application of the load.

For further measurements of residual strengths in order to compare different formulas, the measurement of shear strength after thermal loading was chosen. Shear strength was chosen based on practical experience during the measurement of compressive strength, when the failure of the rollers did not always occur in the same place of the sample, while when measuring the shear strength, the plane of failure of the sample was always the same. The thermal load temperature was chosen at 400 °C in order to achieve more discernible differences when comparing different mixtures, and for the same purpose the heat load times were chosen to be 30 minutes (which achieves complete heating up of the sample) and 15 minutes (half heat load). The list of tested formulas is shown in Table 2.

Table 2.
List of formulas for measuring disintegrability

#	Sand		Binder		Powder additive	
	Type	d_{50} [mm]	Type	Amount [%]	Type	Amount [%]
1	100 % SH32	0.38	EP4158	2.0	WJ4000	0.9
2	100 % SH35	0.20	EP4158	2.0	WJ4000	0.9
3	SH32:SH35 (70:30)	0.33	EP4158	2.0	WJ4000	0.9
4	100 % SH32	0.38	EP4703	2.0	TC4000	1.8
5	100 % SH35	0.38	EP4703	2.0	EP4703	0.9
6	100 % ST54	0.22	EP4158	2.0	WJ4000	0.9
7	100 % MinSand	0.22	EP4158	2.0	WJ4000	0.9
8	100 % Cerabeads	0.22	EP4158	2.0	WJ4000	0.9
9	100 % Chromite	0.35	EP4158	2.0	WJ4000	0.9

The considered formulas included mixtures with different strengths and also with different types of binder system (# 4 and 5). The binder Inotec EP4703 is modified for the purpose of higher resistance to air humidity and was used based on practical experience, when the binder Inotec EP4158 using a different additive than WJ4000 created a mixture of a very thick consistency, similar to dough. This phenomenon is caused by the fact that the binder EP4158 has a lower content of solids and is modified with surfactants based on detergents in order to improve fluidity. The above causes the formation of foam in the process of mixing with the opening material under operating conditions of the mixture preparation (larger amount of the mixture and higher mixing speeds compared to laboratory conditions). The WJ4000 additive contains substances that suppress this formation of foam. Therefore, when testing other powder additives, the liquid component of the binder Inotec EP4703 was used. The results of measuring the shear strength after a given thermal load are shown in the graph in Fig. 5.

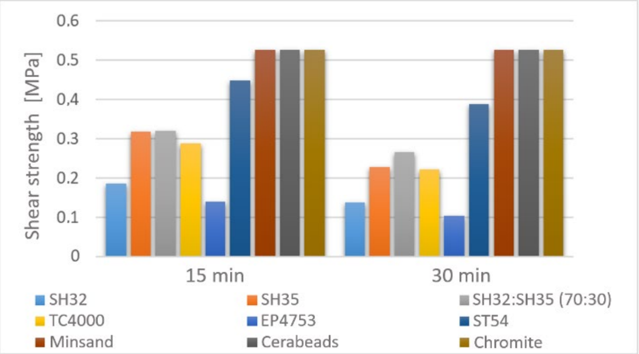

Fig. 5. Results of measurement of shear strength after a thermal load of 400 °C

As it can be seen from the results, the mixtures with a silica opening material have lower residual strengths. The addition of 30 % of fine-grained SH35 to SH32 opening material increases the flexural strength of the mixture, but also the residual strength, which means that the disintegrability of the mixture worsens with the addition of fine-grained opening material. The mixture with the fine-grained SH35 had higher residual strengths than the mixture with coarse-grained SH32, which also supports this claim. The mixture with the angular ST54 opening material had the highest residual strengths of all mixtures with silica opening material, while the flexural strengths are significantly lower. This may be due to the distribution of grains in several fractions and their angular shape may cause higher resistance to shear loads. The lowest residual strengths and the best disintegrability were measured by this method for the mixture using the EP4753 additive. When the samples were loaded for 15 minutes, the residual strengths of all mixtures were higher than after thermal loading for 30 minutes, where the difference between formulas # 2 and 3 was visible. The shear strength was measured in the range of 0.1 – 0.525 MPa with the used strength measuring device, and in this range the samples with non-silica opening materials were not broken. Therefore, this measurement method was evaluated as insufficient for assessing the disintegrability of the mixture, even though it showed a difference in residual strengths with the addition of the fine-grained SH35 opening material to the coarse-grained SH32 opening material compared to the pure SH35 opening material under a thermal load of 400 °C for 30 minutes.

Another proposed method was abrasion measurement on prisms for measuring flexural strength after thermal loading. Abrasion measurement was chosen as another very widespread strength characteristics of sand mixtures, which at the same time, with its mechanism of sample disruption, better simulates the conditions of de-coring of cylinder head castings during hammering by a system of hammers and subsequent disruption and shaking out the remains of the mixture using vibrations. The temperature during loading of the samples in the furnace was chosen at 400 °C based on previous measurements of the residual shear strengths. The time for the prisms to completely heat up to 400 °C was experimentally determined to be 10 minutes by an analogous method of measurement using a thermocouple (type K) in the center of the sample and using a temperature recorder during heating in a resistance furnace heated to 400 °C. The measurement procedure using this method was as follows:

Using a Benetlab laboratory blower machine with settings according to Table 3. Three samples were produced for each mixture listed in Table 2.

Table 3.
Parameters of production of test prisms for measuring bending strength

Core box temp.	Hot air temp.	Blow pressure	Gassing pressure	Exhaust pressure	Delay time for hot air	Hot air purging
180 °C	100 °C	3 bar	2 bar	2 bar	35 s	20 s

Three cooled samples (1 hour, final strength reached) were placed in a preheated resistance furnace (400 °C) with air extraction from the working space of the furnace for 10 minutes during which the furnace was closed. After 10 minutes, the samples were carefully taken out of the oven onto the prepared metal trays, where they cooled for 1 hour at ambient temperature. After cooling, the prisms were sawn in half lengthwise, resulting in 6 shorter samples. Each sample created in this way was weighed to the nearest thousandth of a gram (m_{1i}) precision.

A device made by the GF company was used to measure abrasion on the samples after thermal loading. The sample was placed in the cage of the device. The abrasion measurement lasted 60 s at a constant cage revolution speed of 57 rpm. After stopping the cage, the remainder of the sample was carefully removed and weighed to the nearest thousandth of a gram (m_{2i}). This measurement was repeated for all six samples produced and for each mixture measured.

The term ‘degree of disintegrability’ was defined as a criterion for evaluating the measurement. The principle of calculating the degree of disintegrability consisted in the mathematical expression of the difference in the weight of the sample before and after the measurement in percentage. The higher the degree of disintegrability, the better the disintegration of the mixture. For each mixture, 6 partial results of the weight loss of the sample are obtained, which differ depending on the stay of the sample in the furnace (in the thermal load). The degree of disintegrability is then understood as the arithmetic mean of all measurements calculated according to the equation (1).

$$S_r = \frac{1}{6}.\sum_{i=1}^{6}\frac{m_{1i}-m_{2i}}{m_{1i}}.100 \qquad (1)$$

Where: Sr – degree of disintegrability [%],
m1i – weight of the i-th sample before measurement [g],
m2i – weight of the i-th sample after the measurement [g].

Results for mixtures from Table 2 achieved by using this method are shown in the graph represented on Fig. 6.

It can be seen from the results that the values of the degree of disintegrability correspond to the values of the residual shear strengths, achieved at a load of 400 °C for 15 minutes, with the fact that when measuring the abrasion, it was also possible to observe differences between the formulas with non-silica opening materials. Almost 100 % degree of disintegration was achieved by the mixture with the additive EP4753. According to this method, the mixture with an angular opening material has better disintegration than when measuring the residual shear strengths, which also correlates with the low bending strengths of this mixture. Of the non-silica opening materials, the formula with the Cerabeads opening material reached the highest degree of disintegration. It is necessary to emphasize that the reference thermal load of either rollers or prisms was experimentally determined for a silica opening material. However, non-silica sands have a different specific gravity, heat capacity and thermal conductivity, therefore the results achieved are comparable only if we consider the same heat load. In a real casting, the core is thermally stressed by a higher temperature of the melt and for a longer time (from casting to its complete cooling). That is why a low degree of de-coring in mixtures with non-silica opening materials does not mean worse disintegration. On the contrary, practical experience shows that the cores of water spaces made of artificial opening materials can be de-cored from the casting in the same way as when silica sands are used. In order to achieve comparable results, it would be necessary to determine the time of heating up individual samples with non-silica opening materials rs to 400 °C using a thermocouple (type K) inserted into the sample during heating in the oven. The measurement of the times required to achieve the same thermal load for samples with non-silica opening materials was not carried out in this work.

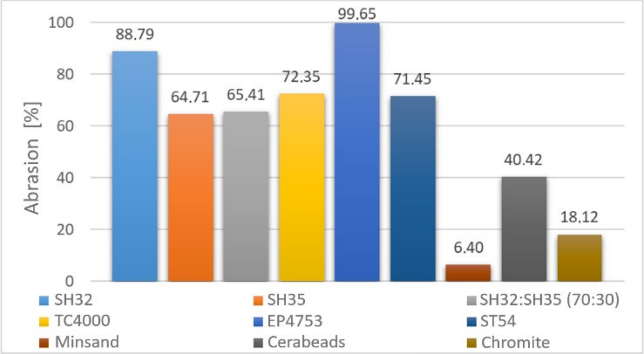

Fig. 6. Results of measuring the degree of disintegration by the abrasion measurement method

2.1. Correlation of the degree of disintegrability and results in real conditions

The subject of this chapter is to verify the correlation between the degree of disintegrability achieved using the created laboratory methodology and real results in operating conditions. For this verification, formulas were proposed for the production of the cores of the water channels of the cylinder head casting, which, in terms of geometric complexity, are the most susceptible to the low disintegration of the mixture when using inorganic binder systems. Table 4 presents a list of proposed formulas suitable for this analysis in terms of core manufacturability.

For the proposed formulas, the degrees of disintegrability were measured in laboratory conditions according to the developed methodology for measuring abrasion after thermal loading of the samples for measuring the bending strength. The results of this measurement are shown in the graph in Fig. 7.

From the measured values of the degree of disintegrability, it is possible to assume different results of de-coring of the water spaces of the casting under operating conditions. The proposed formulas were subsequently used for the production of water channel cores. All castings were de-cored at a temperature of 60 – 80 °C under constant parameters of pressure and hammering time and shaking time on a vibrating unit. After finishing, the spaces of the water

Table 4.
List of formulas for analysis of mixture disintegration in operating conditions

#	Sand		Binder		Powder additive		Hot strength [MPa]	Cold strength [MPa]
	SH32 [%]	SH35 [%]	Type	Amount [%]	Type	Amount [%]		
1	70	30	EP4158	2.3	WJ4000	1.2	2.20	4.60
2	70	30	EP4158	2.4	WJ4000	1.1	2.21	4.40
3	100	-	EP4158	2.4	WJ4000	1.1	1.90	4.20
4	100	-	EP4158	2.3	TC5000	1.3	1.90	3.75
5	100	-	EP4158	2.2	WJ4000	0.8	1.75	3.80

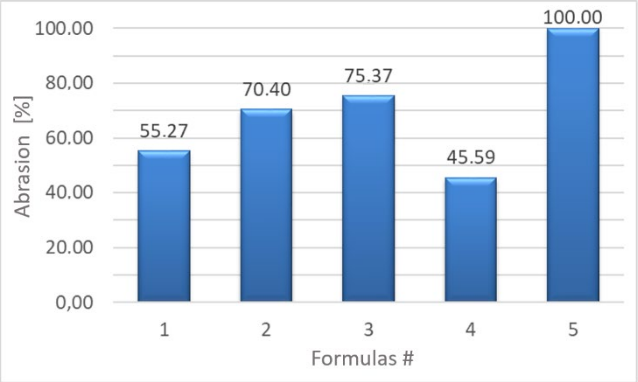

Fig. 7. Results of measuring the degree of disintegration by the abrasion measurement method

channels in the casting were checked using X-ray inspection. Using this method, it was possible to evaluate whether a residual mixture remained in the channels after de-coring. Fig. 8 shows an X-ray image of a casting with a through channel (a) and channels blocked by residual mixture (b).

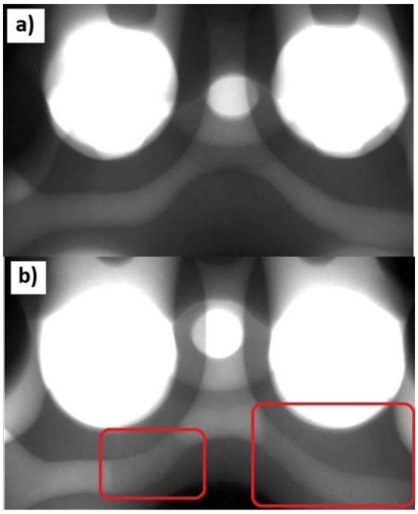

Fig. 8. X-ray images of a casting: (a) With a passing channel; (b) With a blocked channel

The results of the analysis showed that mixture #4 with the lowest degree of disintegrability was not de-cored, castings with cores from mixtures #1 and #2 were de-cored in most cases, but 100 % de-cored castings were achieved using mixtures #3 and #5. The correlation of the degree of disintegrability and real results is shown in Fig. 9.

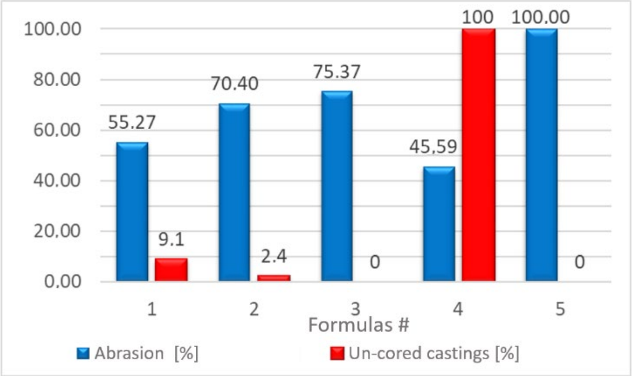

Fig. 9. Results of the disintegration for the mixture in operating conditions

4. Discussion of the results

From the point of view of meeting the minimum specified strength properties and the disintegration of the mixture, formula #3 was the most suitable. Formula #1 had a lower content of liquid component and a higher content of powder ingredient compared to formula #2, which was reflected in the lower degree of disintegrability of mixture #1. This indicates that the mutual ratio of the liquid and powder components of the binder system affects the resulting modulus of the resulting silicate, which further indicates the technological properties of the cores. From the results, it can also be concluded that the type of powder additive used significantly affects the disintegration of the mixture. When using the TC5000 Promotor, the degree of disintegrability was significantly reduced and no casting was de-cored. From this it is possible to conclude that not only the amount (or the mutual ratio of liquid and powder components) of the powder additive, but also its chemical composition significantly affects the modulus and type of the resulting alkaline silicate, which forms connective bridges between the opening material grains and defines the properties of the entire core. By adding 30 % of the fine-grained SH35 opening material to the coarse-grained SH32 opening material, the strength of the mixture increases, but on the contrary, its disintegration after casting worsens. In this case, it can be related both to the higher strength properties of the mixture, but also to the higher Al_2O_3 content in the SH35 opening material (1.266 % for SH32, 2.031 % for SH35), which increases the thermal stability of the binder system and thus worsens the disintegration of the mixture after casting. For the given casting, or formula for the core of the water space, the critical value of the disintegration degree is approximately 75 %. This critical value is significantly influenced by the geometry of the core and its complexity, therefore, in the case of using the proposed methodology for determining the degree of disintegrability using abrasion for a different design of the water core, it would be necessary to determine a new critical degree of disintegrability. From the point of view of the continued development of the created methodology, a coefficient "k" could be created, which would include the complexity of the core (cross-section, number of cross-section transitions, number and position of holes through which the mixture can be removed from the casting during core removal) and could thus be used to calculate critical degree of disintegrability depending on the design of the casting/core according to the formula (2).

$$S_{r\,krit} = 75.k \tag{2}$$

Where: $S_{r\ krit}$ – critical degree of disintegrability [%]
k – geometric complexity coefficient of the core.

For example, k=1 for the most geometrically complex cores (as in the given case), or k=0.9 for less complex cores with thicker water channel cross-sections. To determine the values of the coefficient k, it would be necessary to have a representative sample of the cores of water spaces with different geometric complexity, then experimentally determine their critical degree of disintegrability and find a mathematical dependence for the coefficient k.

5. Conclusions

In this paper, the laboratory results obtained using proposed method were clearly correlated with the results of de-coring under manufacturing conditions, based on which it was possible to determine the optimal setting of the formula for the production of geometrically complex cores of the water space and their complete de-coring after casting. The findings are as follows:

The ratio of the liquid/powder components of the binder system affects the resulting technological properties of the cores. Higher content of the liquid component resulted in lower disintegrability.

Chemical composition of the liquid component considerably influences disintegrability.

Introduction of fine grain sand into the mixture worsens disintegrability of the cores.

Disintegrability is vastly influenced by the geometric complexity of the core and a new formula for critical degree of disintegrability was proposed.

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