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## DETERMINATION OF PARAMETERS OF THE MOULDING SAND RECLAMATION PROCESS, ON THE THERMAL ANALYSIS BASES

### WYZNACZENIE PARAMETRÓW PROCESU REGENERACJI MASY NA PODSTAWIE ANALIZY TERMICZNEJ

For the practical and functional reasons the investigation of the thermal decomposition process is of an essential meaning in relation to the thermal stabilisation of materials and obtaining for them the desired thermal properties. On the other side, thermal tests are carried out in order to identify degradation mechanisms, which is important in the environment protection context, including materials reuse. The cycle of investigations in which thermal TG-DTA methods were applied as supplementary ones for the works on the optimisation of the thermal reclamation process is presented in the hereby paper. The thermal reclamation process as a utilisation method of spent moulding and core sands is more costly than other reclamation methods, but in the majority of cases it simultaneously provides the best cleaning of mineral matrices from organic binders. Thus, the application of the thermal analysis methods (TG-DTA), by determining the temperature range within which a degradation followed by a destruction of bounded organic binders in moulding sands, can contribute to the optimisation of the thermal reclamation process and to the limiting its realisation costs.

*Keywords:* thermal reclamation, organic binders, spent sands, ignition losses, thermal analysis TG-DTA

Ze względów praktycznych i użytkowych zbadanie procesu degradacji termicznej ma podstawowe znaczenie w odniesieniu do problemu procesu stabilizowania termicznego materiałów i otrzymywania dla nich pożądaných właściwości termicznych. Z drugiej strony badania termiczne prowadzi się w celu rozpoznania mechanizmów ich degradacji, co ma też znaczenie w kontekście ochrony środowiska, w tym ich powtórnego wykorzystania materiałowego. W publikacji przedstawiono cykl badań, w którym zastosowano metody termiczne (TG-DTA) w uzupełnieniu do prowadzonych prac nad optymalizacją procesu regeneracji termicznej. Proces regeneracji termicznej jako sposób utylizacji zużytych mas formierskich i rdzeniowych w stosunku do innych rodzajów regeneracji zużytych mas jest bardziej kosztowny, ale jednocześnie dający w większości przypadków najlepsze oczyszczenie osnowy mineralnej ze spoiw organicznych. Stąd zastosowanie metod analizy termicznej (TG-DTA), poprzez określenie zakresu temperatury, w którym dochodzi do degradacji, a następnie destrukcji związanych spoiw organicznych w masie, może przyczynić się do optymalizacji procesu regeneracji termicznej i zmniejszenia kosztów jego realizacji.

### 1. Introduction

A reclamation process temperature constitutes an essential factor, deciding on a binder efficient removal in the thermal reclamation of spent moulding sands. Since the thermal reclamation process requires high investment and usage expenditures, performing investigations leading to limiting its realisation costs seems justified. One of such operations is a determination of the minimal temperature range of the thermal reclamation for the given moulding sand with an organic binder. A large variety of foundry resins, offered for preparations of moulding and core sands requires an individual estimation of their utilisation possibility. The thermo-gravimetric and differential thermal analysis (TG-DTA) [1, 2] can be helpful. This method allows to determine the temperature range within which a degradation followed by destruction of an organic

binder in a moulding sand occurs [3]. Having such information and using properly constructed devices, it is possible to create individual processes, which will be efficient, in removal of thermal degradation remaining products and binder left-overs from the grain matrix, as well as economical due to limiting the energy consumed in its realisation [4-7].

Another important element influencing the thermal reclamation costs is the construction of the device, in which a grain matrix purification is performed in the determined way. In order to find out which factors decide on the process pathway the experimental thermal reclaimer was built [8, 9]. Various possibilities of the thermal reclamation process are created in this specially designed device. The reclaimer chamber is equipped with a few heat sources situated in different places. Various conditions of mixing (either by air of ambient temperature or by air warmed by combustion gases in a recuperator)

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the moulding sand being reclaimed were also created (simultaneous, sequential). The process pathway is monitored, reclamation temperature is set (e.g. according to the data obtained from the thermal analysis) and controlled in the required range, and the work parameters are recorded [8-10]. The collected information will be used as guidelines for the determination of the most optimal realisation of the thermal reclamation process.

## 2. Methodology

### Materials

Organic binder: urea-formaldehyde resin modified by furfuryl alcohol, FR 75A – 1.0 part by mass.

Hardener: mixture of organic (from sulphone group) and inorganic acids, PU-6 – 0.5 part by mass.

Matrix: high-silica sand Jaworzno-Szczakowa – 100 part by mass.

### Thermal analysis

Thermal investigations were performed by means of the thermal analyser NETZSCH STA 449 F3 Jupiter®, which allowed simultaneous measurements of TG and DTA. This warranted obtaining independent signals recorded under the same measurement conditions, it means at the same temperature increase rate (10°C/min), the same atmosphere and gas flow rate (40 ml/min). Measurements were carried out in the oxygen (air) and oxygen-free (argon) atmosphere. Samples subjected to the TG-DTA analysis were of a mass of approximately 15 mg. Platinum crucibles, which enabled measurements up to 1000°C, were used.

## 3. Thermal analysis

The series of TG curves for the binder sample, in a form of hardened furan resin in the mixture of organic acids from a sulphone group and inorganic acids, recorded in the oxygen and oxygen-free atmosphere, are presented in Figure 1. It can be stated, on the bases of the obtained TG curves, that the resin degradation pathway depends on conditions of the decomposition process, which can supply an essential information for the determination of conditions for performing the thermal reclamation of spent sands. During the thermal decomposition in the oxygen atmosphere two endothermic effects (at 101°C and 342°C) and two exothermic effects, the most probably related to sample burning at temperatures of 617°C and 720°C, were seen in the DTA curves (Fig. 1a).

During the decomposition in the oxygen-free atmosphere two effects: endothermic (86°C) and exothermic (280°C), can be observed in the DTA curves. In addition, up to a temperature of 1000°C, the sample was not completely decomposed and the remaining part of the mass was above 48% (Fig. 1b).

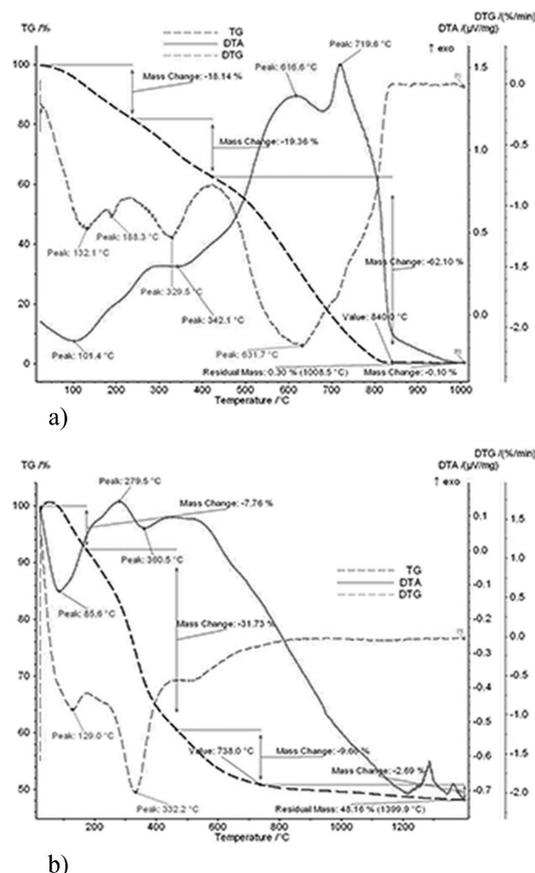


Fig. 1. TG-DTA curves of the resin obtained in: a) Oxygen atmosphere, b) Oxygen-free atmosphere

## 4. Analysis of binder mass losses

Ignition loss measurements constitute another method of determining a thermal destruction of bounded binders. The determined amount of a moulding sand is placed in the furnace heated to a temperature at which the complete burning (in the oxygen atmosphere) of the majority of organic compounds occur (950°C). Individual carbon derivative substances can undergo a destruction at various temperatures. The required temperature range can be determined by means of applying ignition losses for individual temperatures – to the moment when the sample mass loss stabilises. If we assume that bounded binder constitutes 1.48% of the moulding sand and that this is 100% of the total amount which we would like to remove from the spent moulding sand, the performed ignition loss estimation at individual temperatures recalculated in relation to the bounded resin total content, can be presented in a form of a binder mass percent losses. This is illustrated in Fig. 2. Above a temperature of 700°C there is a lack of losses of the bounded resin, which indicates that the burning process of organic substances is finished. The obtained results were combined with mass losses of the bounded binder presented in TG curve. Discrepancies in the presented data are the result of several factors:

– Thermo-gravimetric analysis was performed on the bounded binder alone, since in the whole volume of 15 mg sample there was 100% of materials subjected to a thermal destruction;

– Investigations of ignition losses were realised on 30 g samples of spent furan moulding sand, crushed and forming a porous structure, in which an easier air contact facilitates a burning process, simultaneously only 1.48% of a total sand undergoes a thermal destruction;

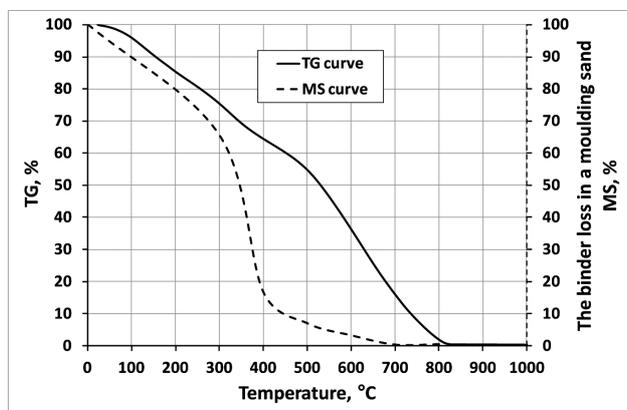


Fig. 2. Diagram of the bounded binder loss (TG curve) and the binder loss in a moulding sand, in dependence on a temperature

– A temperature influence on the investigated materials was different. In the thermo-gravimetric analysis the temperature increase was carried out with a constant rate of 10 degrees in a minute (this was a continuous recording), while in case of ignition losses the samples were placed into the furnace heated to the determined temperature and kept there for 1 hour.

The combination of the results of two different methods of investigating the thermal destruction (analytical and technological) of the same binder, created – in the common diagram – the zone in which the minimal temperature required for the moulding sand reclamation should be searched for.

Significant differences in percentage amounts of the remaining binder depending on the applied destruction technique can be seen in Figure 2. At 400°C and 500°C the difference is 50%, while at 600°C the values differ by 35%. At a temperature near 800°C both results indicate the same destruction level of the bounded binder.

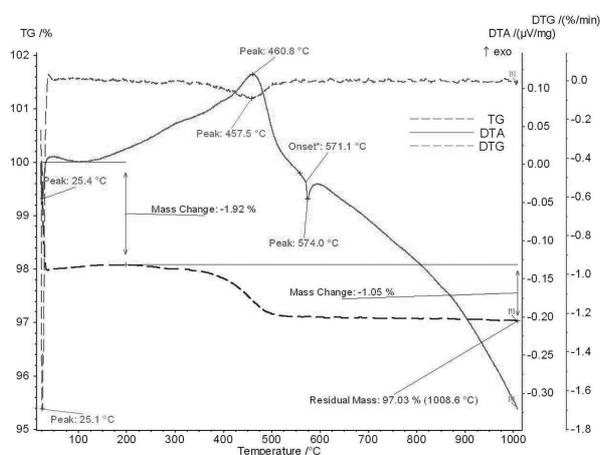


Fig. 3. TG-DTA curves of the spent moulding sand with organic binder, obtained in the oxygen atmosphere

For more complete illustration of the problem, we performed the thermo-gravimetric analysis of the spent furan mass, being fully aware that 15 mg sample is not representative

from the point of view of the amount of the spent binder on grain surfaces. However the bounded resin, contained in the sample, should be burned at strictly determined temperature and under conditions being nearer the actually realised thermal reclamation process (binder – grain matrix). The obtained results are presented in Figure 3.

The significant change of the amount of the moulding sand sample in the temperature range 400-500°C, while above this temperature quite insignificant changes, are observed and shown in Figure 3. Thus, comparing three performed tests, it can be assumed that an efficient thermal reclamation is expected in the temperature range: from 600 to 700°C.

## 5. Realisation method and the obtained results analysis

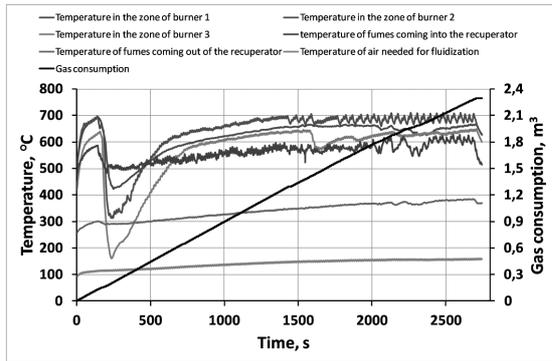
The spent moulding sand, after hardening according to the composition given above, was crushed in the jaw crusher and then sieved through 0.8 mm sieve. Such material was subjected to thermal reclamation procedures. The spent moulding sand from the no bake process was charged into the reclamation chamber after its heating to the required operation temperature and obtaining the air fluidisation temperature of 100°C. The spent sand (charge: 10 kg) was reclaimed at temperatures: 500, 600 and 700°C, with sequential mixing of the material layer (zone 1-5 seconds, zone 2-5 seconds, zone 3-5 seconds). During the reclamation process (every 5 minutes) small portions of the reclaim were taken, via the hole for pouring out, to determine their ignition losses. At the reclamation temperature of 700°C, the sampling process was carried out in two versions: after the sand obtained the required temperature in the middle part of the device and from the moment of charging the reclaimer with the spent furan sand. At the maximum temperature, assumed in investigations, the reclamation cycle, during which the sand layer was mixed by the air from surroundings (cold), was performed.

Some examples of the recorded parameters of the reclaimer operation, performed at a temperature of 700°C, are presented in Figure 4.

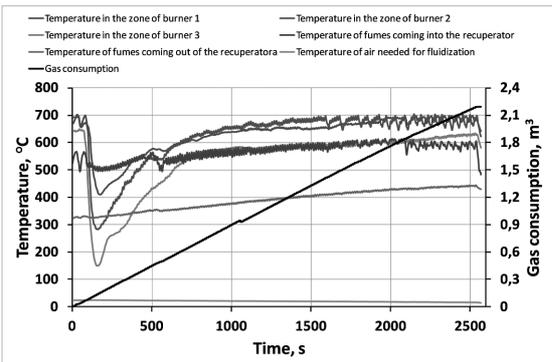
The reclamation temperature of 500°C of spent furan moulding sands is not sufficient for a complete matrix cleaning from a binder (Fig. 5). A temperature increase by 100°C, intensifies the destruction process of the applied resin, but does not clean fully the matrix grains. The best cleaning is achieved when the process is carried out at a temperature of 700°C, which is indicated by the ignition loss values (Fig. 5). It can be also noticed that, while in between 500°C and 600°C differences in ignition losses for individual reclamation times are significant – while in the next temperature range these values do not differ a lot, apart from the shortest time – being 5 minutes.

The presented above results of ignition losses concern the situation when sampling started at the moment when the middle part of the reclaimer chamber obtained the set temperature. Thus, the real time of the operation is much longer since a spent moulding sand is already reclaiming during the sand layer heating to the set temperature. Differences in ignition loss values, in dependence on the assumed process beginning, (from the moment of the reclaimer charging and from the mo-

ment when the reclaim together with spent sand achieved the set temperature), are presented in Figure 6.



a)



b)

Fig. 4. Recorded temperatures and gas consumptions during reclamation procedures, at a temperature of 700°C: a) The sand layer mixed by the air from the recuperator, b) The sand layer mixed by the air from surroundings

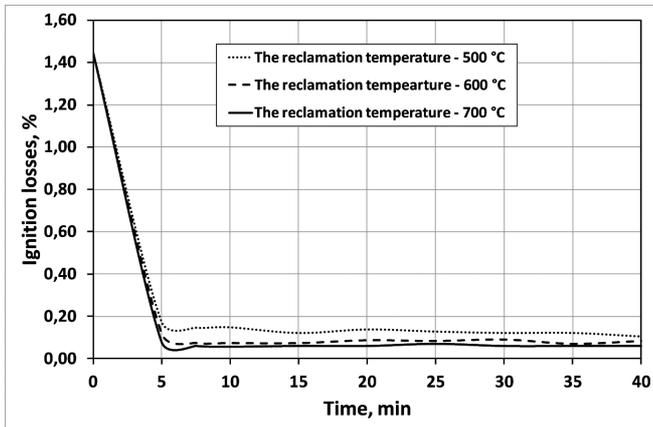


Fig. 5. Ignition losses of the reclaim in dependence of the reclamation time, in the thermal reclaimer with sequential mixing of the material layer, at various temperatures of the process

On the bases of the obtained results (presented in Figure 6) it was found that, during the initial period of the process a part of the supplied energy (from gas burning) is used for the compensation of differences in temperatures of the charge (Fig. 4) and the reclaimer chamber, which was cooled after an introduction of the spent moulding sand of an ambient temperature. Ignition loss values, presented in Figure 6, allow to state that the total time needed for the effective cleaning of the grain matrix from a spent binder, realised in the device of the given work parameters, should be 20 minutes.

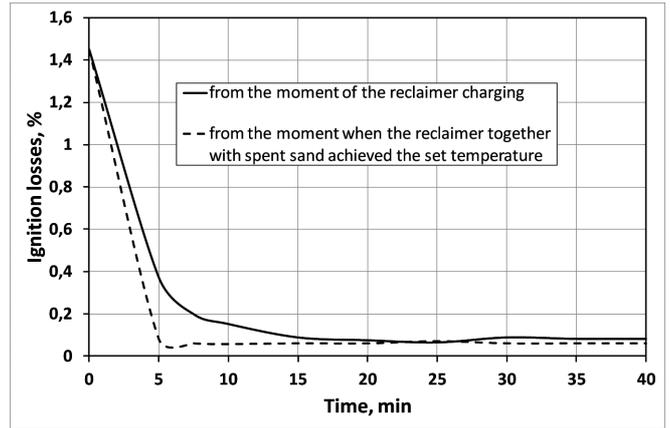


Fig. 6. Ignition loss values of the reclaim in dependence on the reclamation time, in the thermal reclaimer with the sequential mixing system of the sand layer – for various sampling ways

The analysis of factors influencing the efficient thermal reclamation of the spent moulding sand with furan resin in the experimental thermal reclaimer, allowed to determine the basic process parameters. The following parameters were assumed: temperature: 700°C, time: 20 minutes, sand layer mixing by warm air.

The moulding sand was prepared on the bases of the reclaimed material obtained by the thermal reclamation, according to the given above recommendations. The moulding sand of the same composition but with the fresh grain matrix, was also prepared.

Shaped samples for testing the bending strength (after hardening) were made from both moulding sands. The obtained results, comparing the bending strength of both moulding sands, are presented in Figure 7. In the investigated case, the moulding sand on the grain matrix obtained in the thermal reclamation process was characterised by a higher bending strength than the moulding sand prepared on the fresh sand.

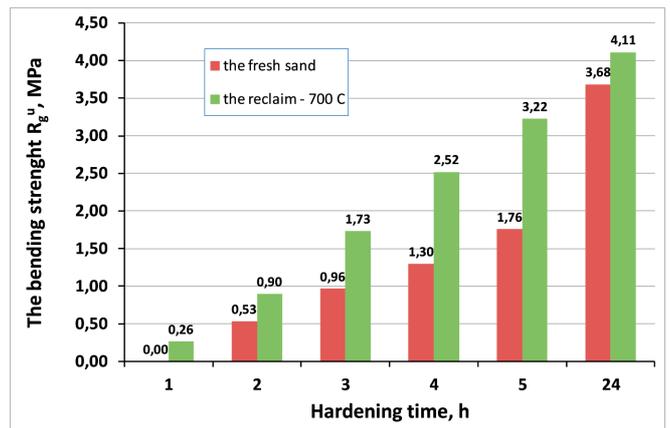


Fig. 7. Comparison of the bending strength of hardened samples made of the fresh sand and of the reclaim

## 6. Conclusions

The performed thermal analyses (TG/DTA) confirm the suitability of the method for selecting the thermal reclamation temperature. However, it appeared that the thermal degradation pathway depends on the composition and form of the tested

material. Tests of the hardened organic binder alone allow to determine the maximum temperature, which warrants the total binder destruction. However, in case of the spent moulding sand sample (binder – quartz matrix) the binder destruction process occurs at lower temperatures. This discrepancy results from a form of the tested sample. Spent moulding sand subjected to thermal analysis constitutes the system composed of fine matrix grains covered by a thin binder envelope, forming a porous structure, which allows a free air access into the whole sample volume. The presence of an oxidising atmosphere around spent moulding sand grains is essential in relation to the progressing degradation of the sample – being the compacted bounded resin – since it facilitates the destruction of a thin binder layer. How important is the presence of an oxidising atmosphere indicates the thermal analysis of the hardened resin under oxygen-free conditions (argon atmosphere), where at the set temperature range, the binder was not totally destructed and 48% of the sample remained. The obtained results confirm and simultaneously indicate that costs limitations of the thermal reclamation can be searched for in the way of the process realisation. High temperatures are not sufficient factors of the process efficiency. The application of the proper way of mixing of the spent moulding sand layer, determined width of the processed layer, amount of the supplied air and its temperature, are elements which can have a significant meaning for the development of the economic (limiting costs) performance of the thermal cleaning of matrix grains from a spent binder. The carried out strength investigations

confirm that the reclaim is the most suitable material for the preparation of moulding sands.

#### Acknowledgements

This work was carried out with a financial support of the National Committee for Scientific Research No N N507 513139.

#### REFERENCES

- [1] B. Grabowska, M. Szucki, J.Sz. Suchy, S. Eichholz, K. Hodor, *Polimery* **58** (1), 36-41 (2013).
- [2] K. Pielichowski, J. Njuguna, Thermal degradation of polymeric materials, Rapra Technology, United Kingdom, (2005).
- [3] B. Grabowska, *Archives of Foundry Engineering* **10** (2), 99-102 (2010).
- [4] M. Łucarz, *Metalurgia, Metalurgija* **45** (1), 37-40 (2006).
- [5] R. Dańko, *Arch Metall Mater* **58** (3), 809-812 (2013).
- [6] S.M. Dobosz, P. Jelinek, K. Major-Gabryś, *China Foundry* **8** (4), 438-446 (2011).
- [7] M. Brzeziński, A. Stawowy, R. Wrona, *Arch Metall Mater* **58** (3), 867-870 (2013).
- [8] M. Łucarz, *Arch Metall Mater* **58** (3), 923-926 (2013).
- [9] M. Łucarz, *Archives of Foundry Engineering* **12** (1), 125-130 (2012).
- [10] M. Brzeziński, *Arch Metall Mater* **55** (3), 763-770 (2010).