



Behaviour of pellets from secondary raw materials when charging in bath-melting reactors

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Abstract

Due to the rising copper demand the recovery of copper from extremely complex and low copper containing materials like fine-grained dust and sludge will become more important in the future. Today these materials are often used as material-mixtures with classical scrap in recycling-processes [1].

Copper recycling processes consists increasingly of bath-melting technologies like the ISA-Smelter. Due to the thermal updrafts and the flue gas stream of such a furnace there is the danger that the fine-grained materials swirl up during or shortly after charging. In worst case they don't get into the bath but will be carried out of the furnace through the thermal of the flue dust stream. Copper losses and adverse effects to the overall process are the consequences [5]. An agglomeration of these fine-grained materials to pellets can avoid these [2].

Within this research work the behaviour of such secondary raw material pellets was investigated analogue to the charging procedure into a bath-melting reactor. In a first step the behaviour of pellets in contact with hot gases was investigated by measuring changes of their weight and form. A second step concentrates on the hit of the pellets onto the melt. The mechanical stability, the maximal residence time and the grade of metallization of the pellets were in the focus of the experiments.

In this paper the test facilities and methods of the experimental works as well as the evaluation of the results will be presented.

1. Background and task

The high demand for copper, in particular from China, India and Russia, has forced up in the past the copper price and at the same time has limited the availability of easily recyclable copper scrap with middle and high copper contents at the market. Considering this background western secondary copper producers have been forced to use extremely complex and low copper containing materials, together with classical copper scrap, as a secondary material mixture in their recycling processes [1][4][7].

Nowadays increasingly bath-melting reactors are used in secondary copper smelters, for instance ISA-/Ausmelt reactors. These reactors have in the recycling process many economic and ecological advantages. They are very adaptable concerning the kind and the humidity of the input material, allow an excellent material mixing due to high bath turbulence and reach high rates of melted material with low emissions of dust. Using a batch-mode blister copper can be directly produced. The reduction and converting step, which are usually takes place in different furnaces, can be combined in one unit only [3].

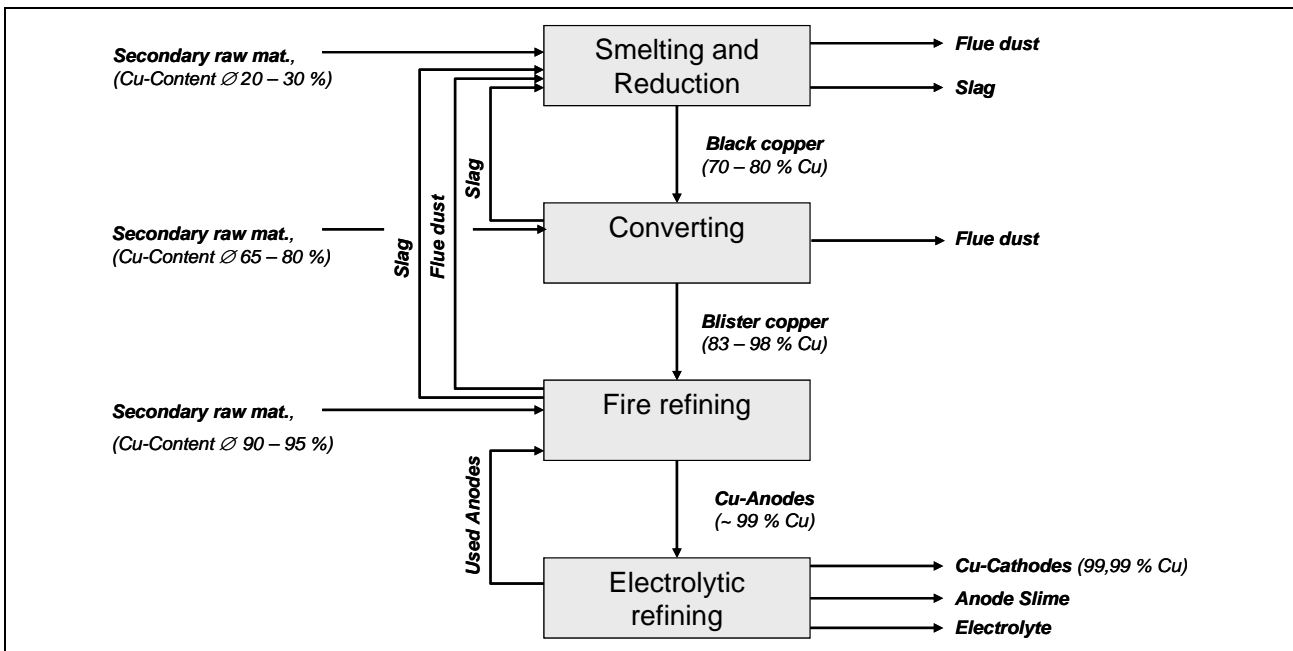


Figure 1: Flowchart of copper producing from secondary raw materials [6, adapted]

However, the use of dust and materials with very low grain size represents special challenges to the processing. The good mixing of the melt by generation of big bath turbulence increases on the one hand the reaction rates within the processes; on the other hand huge thermal up winds are generated. Fine-grained and dust-shaped materials can be thereby whirled up already during or shortly after charging. In the worst case this particles reach not the bath, but are carried out of the furnace by the thermal of the flue gas stream [5].

As a result of this effect metal losses results which can cause at the same time negative effects on the whole process. Sticking of already melted particles in the exhaust gas outlet of the reactors or in units downstream, as for example waste heat boilers, carries the danger to disturb the flue gas streaming of the overall system. Filter systems must be designed more generously, possibly a higher circulation of material is necessary. However, such negative effects can be avoided or at least decreased by a pelletization of the fine-grained input materials [2].



Nevertheless, the success of such a procedure depends on the stability of the pellets compared with a spontaneous heating. Beside the determination of a minimum needed amount of binding agent the drying time of the pellets is very important.

In the following the method to investigate the behaviour of such pellets from copper-containing secondary raw materials, which are falling through a hot furnace atmosphere and hitting a moved slag surface will be presented. Afterwards the experimental results and their assessment will be shown [5].

2. Materials and preparation

Three types of different materials with various copper contents, which are already used as secondary raw materials in the copper recycling, were investigated.

The materials were characterised first concerning their suitability for pelletizing, their grain size distribution as well as their thermo-chemical behaviour. Afterwards pellets were produced from these materials. Depending of their drying time and used binding agent, the produced pellets were investigated concerning their pressure- and abrasion-resistance.

2.1 Used materials for the trials

The material mixtures (see figure 2) were from 22 to 25 different secondary materials in different shares. About 25 % of each material mixture consisted of return material from the anode furnace process and galvanic sludge. The material mixtures had all only a poor copper content, which varied from 17.5 % (material C) to 30.3 % (material A).

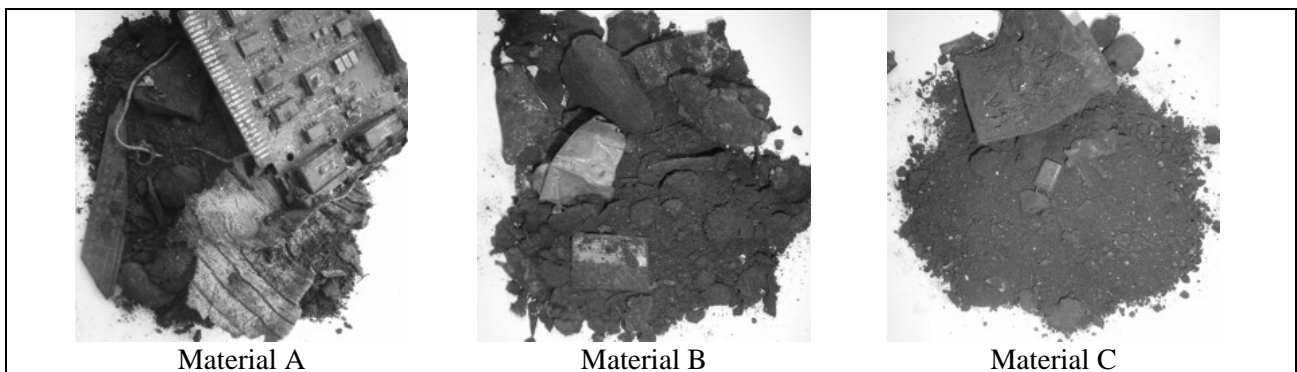


Figure 2: Used secondary raw materials for the experimental works [5]

Because these material mixtures were unsuitable for pelletizing, a screening was done with a maximum grain size of 4 mm. The change of the material composition had to be accepted. The screened material was the source material for the production of the pellets. The following diagram shows the results of the chemical analyses for the raw material as well as the screened material. It was striking



that by the screening process with all three material types a reduction of the copper content has taken place by rise of the SiO₂ content at the same time.

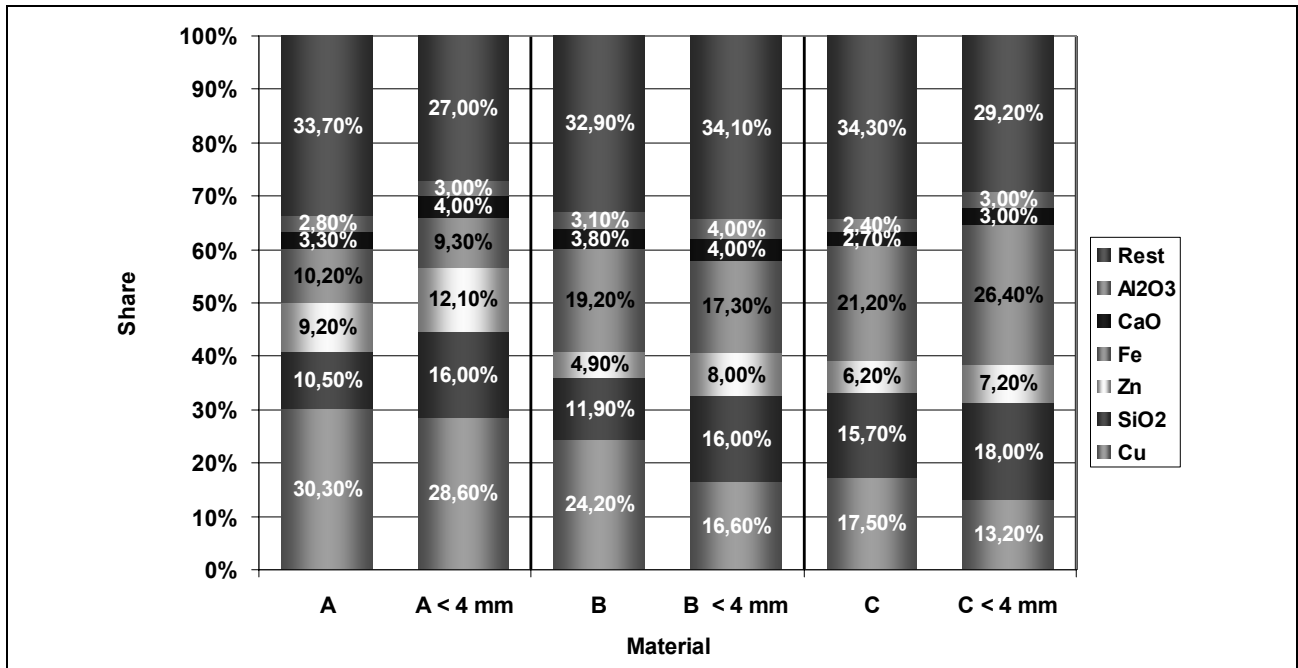


Figure 3: Composition of the material mixtures before and after the screening process [5]

The following diagram shows that after the screening process the grain size distribution of all three materials is very wide. Regarding the pelletizing process this is a positive aspect, but the maximum grain size of 4 mm as well as the high share of grains bigger than 1 mm has a negative influence on the pelletizing process.

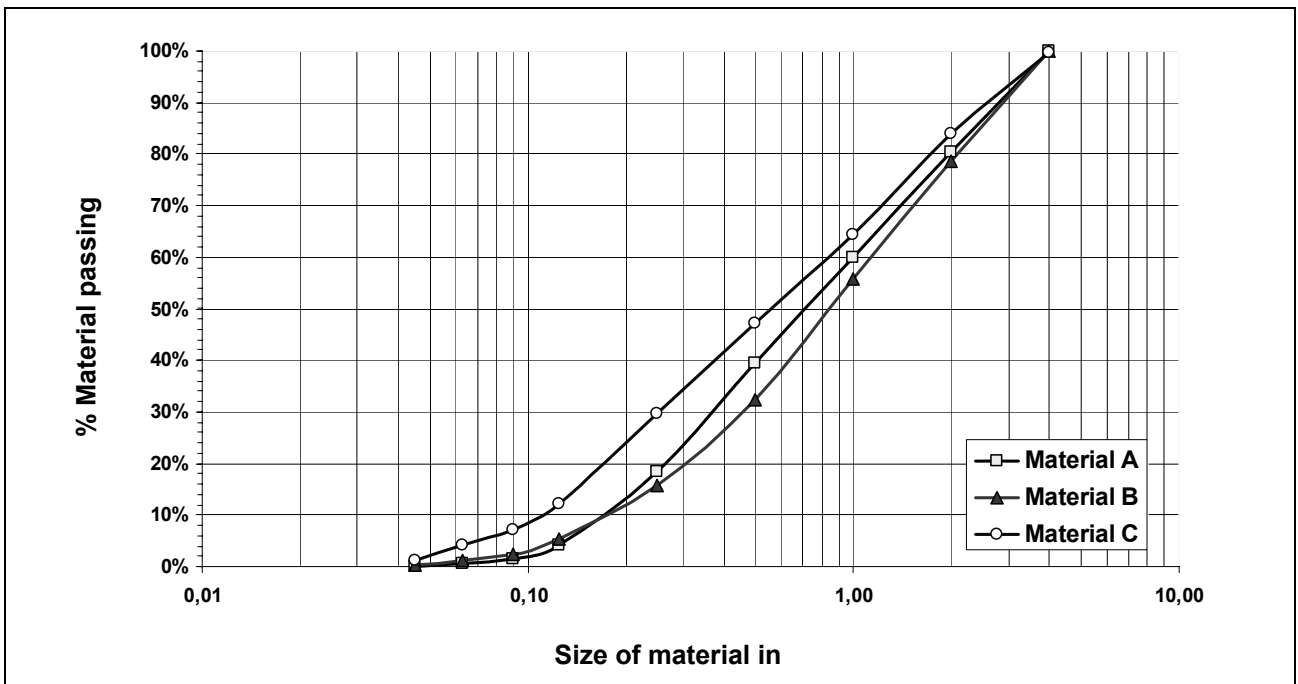


Figure 4: Grain size distribution of the three material mixtures after screening [5]



Material A and B are relatively coarse in comparison to Material C. This becomes clear in the d50 values of the grain size distribution. Material C shows a value from $d_{50} = 0.55$ mm, material A from $d_{50} = 0.7$ mm and material B from $d_{50} = 0.85$ mm.

2.2 Agglomeration of the materials

For the pelletizing of the material mixtures a pelletizing dish in laboratory scale was available. The diameter of the pelletizing dish was 50 cm with rim of 11 cm height. The rotation speed was adjustable by variable from 9 to 51 rpm. The plate inclination was 45° , an inclination which is also usual in the industry for optimum operating conditions.

Due to the inhomogeneity of the materials it was not possible to carry out the pelletizing in the small scale under stationary conditions. Thus no constant values could be put with regard to the added amount of liquid, the rotating speed of the pelletizing dish or duration of the pelletizing process. The added amount of liquid differed between 0.25 and 0.5 l, the rotating speed of the pelletizing dish between 10 and 15 rpm.

Before the planned experiments starts suitable binding agents had to be found first. These should fulfil at least two aspects:

- uncomplicated production of spherical pellets with an diameter of 10 to 13 mm
- sufficient pressure resistance for the experimental works

Investigations of different binding agents shows that 1% of Portland cement as well as 1% of burnt lime as a binding agent addition to the three material mixtures fulfilled the above mentioned aspects. Therefore in total six different types of pellets were available for the experimental works, which are investigated concerning other properties, e. g. like pressure resistance and abrasion as a function of the drying time.

The following set of pictures shows very well that the screened material mixtures contents also long metallic objects, mostly thin wires, which were integrated into the pellets.

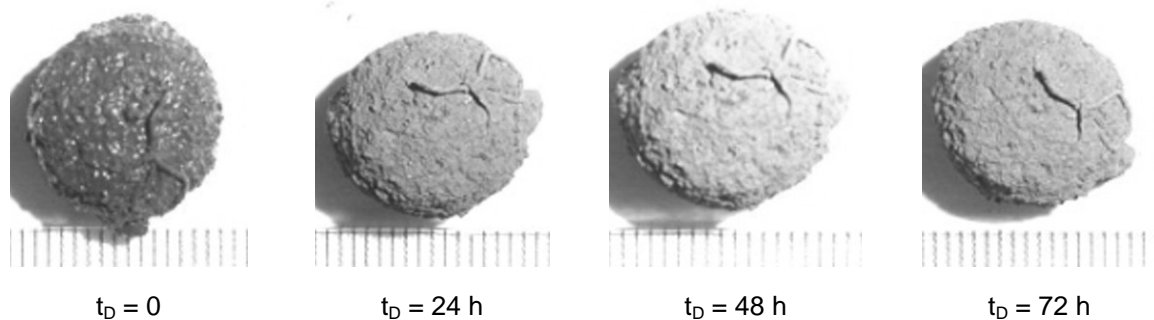


Figure 5: Shape of a pellet during the drying time [5]



The integration of such metallic particles in the pellets is positive, to avoid metal losses due to the turbulent conditions in a bath melt reactor. However, it can at the same time cause also instability of the pellets.

The following diagram shows that with all types of pellets a clear rise of the pressure resistance enters after 18 hours drying time. For nearly all pellets which are produced of the coarser materials the maximum pressure resistance is reached after 24 hour drying. Nevertheless, with increasing drying time the loading capacity decreases again.

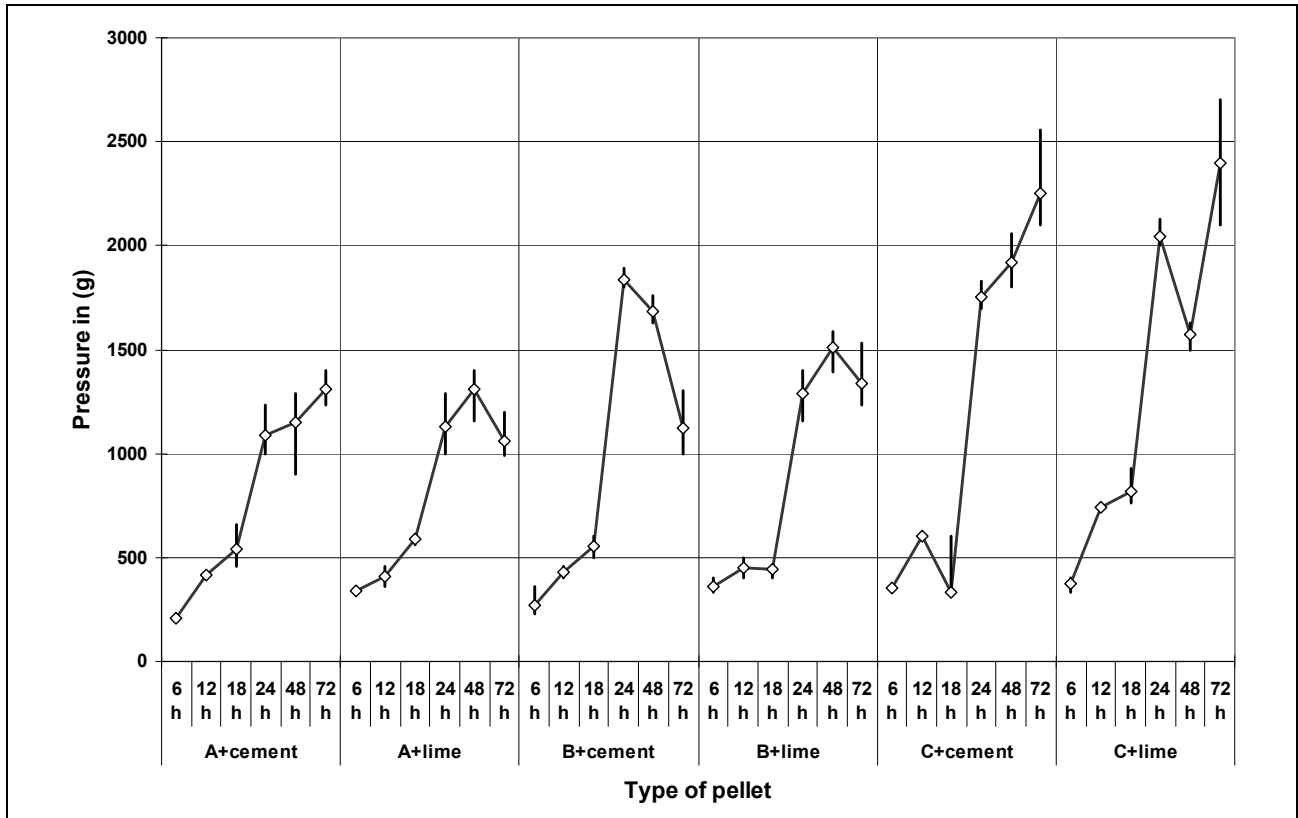


Figure 6: Pressure resistance of all six pellets times due to the drying time [5]

From the following diagram a correlation is to recognise between the grain size distribution of the materials and the values of abrasion. It is evident that the share of the abrasion rise with increasing amount of coarse particles in the material mixtures. The abrasion of the pellets from the fine material C is about nearly all drying steps steadily still approx. 4%, the abrasion of the pellets from the coarse material A lies already between 4 and 7% and for pellets from the most coarse material B with 6 to partial 11% again clearly higher. As a rule the abrasion increases with increasing drying time of the pellets and coarser basic material. A small average particle diameter of the basic material has a positive effect on the surface state as well as the binding of single particles in the pellets itself, so that material losses are decreased as a result of the abrasion.

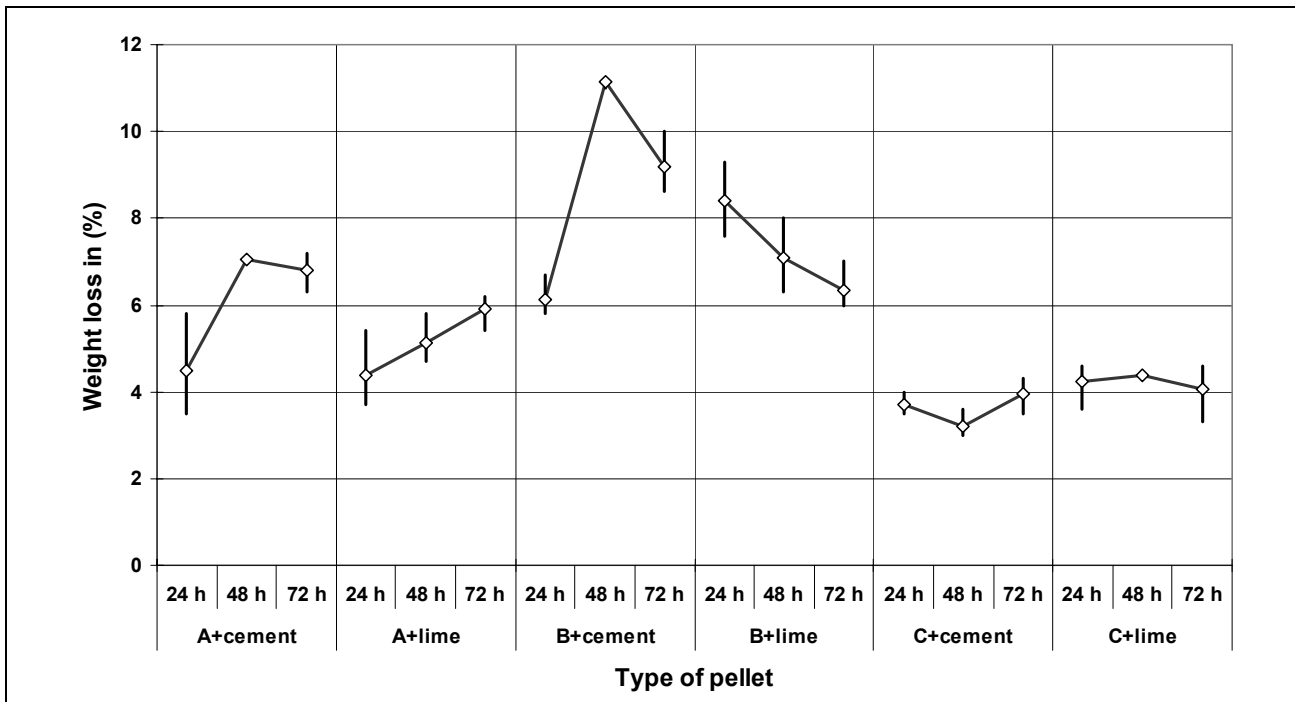


Figure 7: Abrasion of all six pellets times due to the drying time [5]

3. Experimental procedure

3.1 Test facilities for spontaneous gas heating trials

The aim of this experimental step was to investigate the change of mass and shape of single pellets for different temperature steps according to the temperature profile inside a bath melting reactor. For the investigations a Tammann furnace was used. The measurements were done by a precision scales, a special camera system as well as a measuring computer for the data admission.

The furnace space is accessible from above and below. For the weight measurement the scale was installed above the furnace. A hook under the scale allows the pellets hanging in the furnace. The mass change was online recorded. The special camera system was installed below the furnace and the pellets were observed through a small window, which was integrated in the lower furnace end of the Tammann furnace.

For the online measurement of the geometrical behaviour of the Pellets by means of video technology, the window was continuously cleaned by means of argon from falling dust particles and parts of the pellets. Besides, the pellets were positioned by means of a stiff hanging in the hot furnace area to receive sharp video recordings. The overall system, consisting of measuring equipment and the furnace system, is shown in the following picture. In the left area of the picture the hanging device at the hanging position of the single pellets with a centring device arranged above the furnace opening is shown.

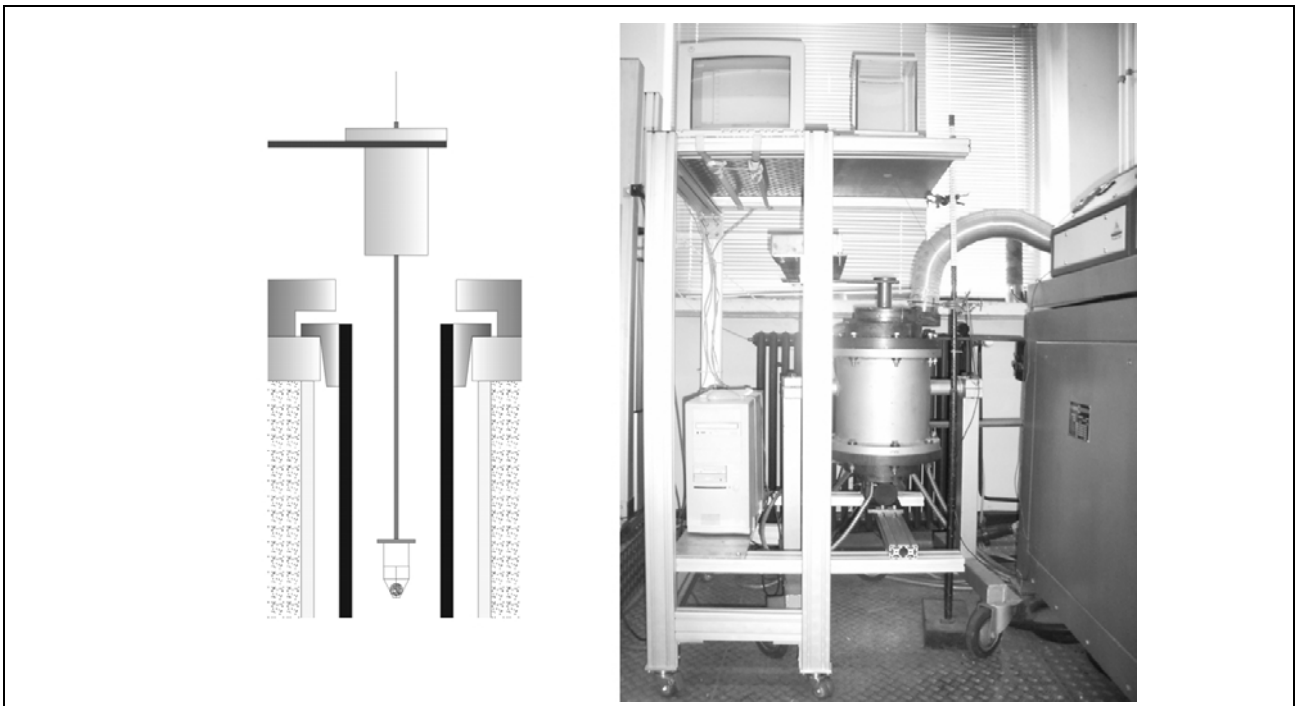


Figure 8: Overall system for the experimental works (right) and hanging device and position of the pellets in the furnace (left) [5]

3.1.1 Test parameters

During the fall down of a pellet through the furnace space of an ISA-Smelter from the charging opening to the melted bath different temperature zones will be passed. For the trials pellets with the parameters drying time, type of binding agent as well as material type were investigated at temperatures of from 900 to 1150 °C in 50 °C to steps. The aim was, to position the pellets as quickly as possible into the hot zone of the furnace at the hanging position and to measure their weight change

The free fall of a single pellet through the furnace space of an Isa-Smelter lasts according to the construction height of the furnace maximum 2 seconds. Nevertheless, in the reality opposing forces counteract against the free fall. The frictional forces of the gas contained in the furnace space as well as lifting forces by the turbulent thermal in the furnace space work against the falling movement. In the upper area of the furnace suction forces from the exhaust gas system can have, in addition, also an influence on the falling time of a pellet. It was estimated within the scope of this work that a pellet needs in the reality up to the contact with the slag phase max. 5 seconds. Nevertheless, the test time became four times more to have at sufficient data amounts for the evaluation of the processes in a pellet at different temperatures.

The following table shows the used test parameters.



Table 1: Parameters of the trials

	Change of weight	Change of shape
Drying time	72 h, 48 h, 24 h, 18 h, 12 h, 6 h	24 h, 6 h
Temperatures	900 – 1150 °C in 50 °C steps	
Atmosphere	Argon	
Material	A, B, C	
Binding agent	1 % burned lime 1 % Portland cement	

3.1.2 Evaluation Method

Following properties of the pellets as a function of drying time, binding agent as well as temperature were shown:

- Time and number of destroyed pellets during and after hanging into the hot furnace
- Weight lost of the pellets until reaching their hanging position
- Weight lost of the pellets during the first 20 seconds after reaching their hanging position

The mass change was continuously observed during the trials by the on-line measurement. Per temperature step the weight changes of 5 Pellets of the same kind was recorded per second and was written in a data bank.

For the evaluation of the results for every pellet the first measuring point which shows the reaching of the hanging position was identified manually. The weight change of each pellet was shown from this first measuring point for a time of 20 seconds in a graph. For each type of pellet and for each temperature step the resulted graphs were transferred into a diagram and average graph was generated. The variables of the equation of the best-fit line of this average graph characterise the measure of the decrease in weight before and after reaching of the hanging position of the pellets in the furnace.

The following picture shows a screen shot of the used programme for the weight measurement. The weight change of the pellets with increasing time of exposure is recognizable.

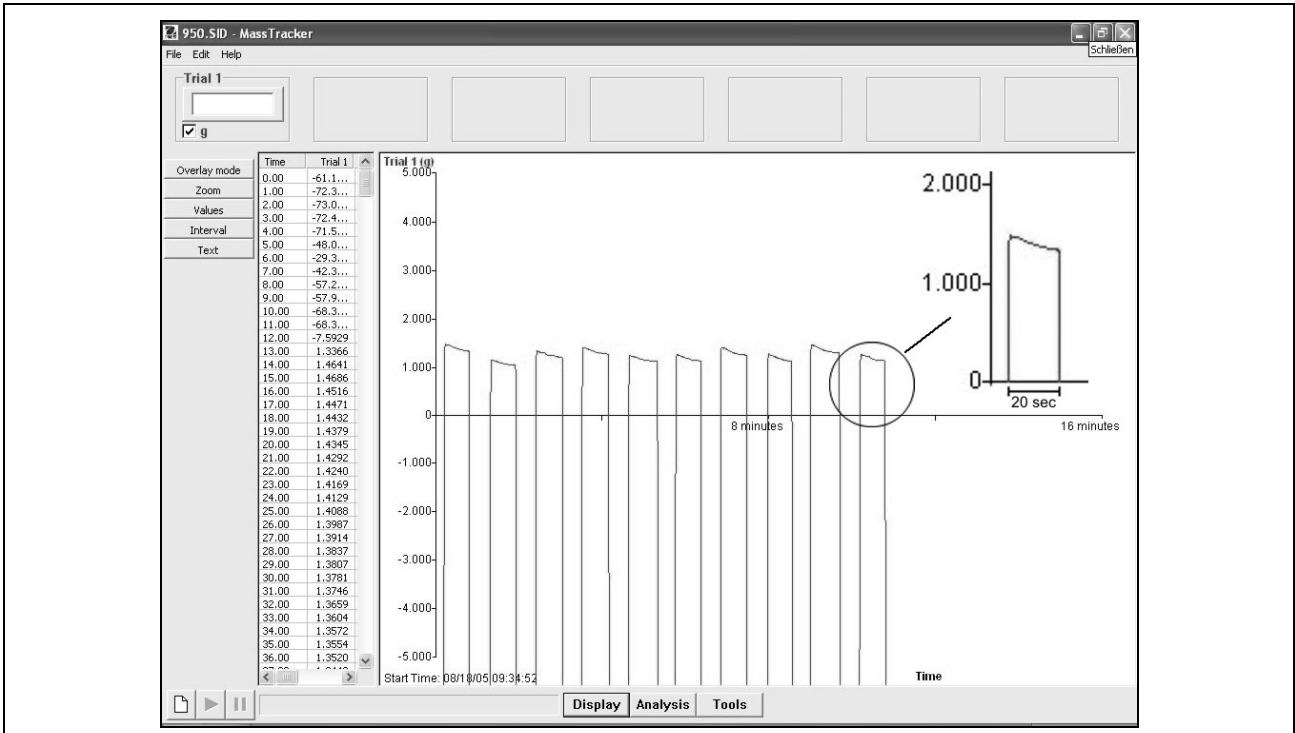


Figure 9: Screen shot of the weight measurement programme [5]

The following picture shows exemplarily for pellets from material B under use of lime as a binding agent an evaluation diagram. The variables of the linear equation from the best-fit line of the average graph shows on the one hand the weight of the pellet when it reaches the hanging position (starting point of the automatic weight measurement), and on the other hand the decrease in weight from this starting point of the weight measurement for the next 20 seconds (gradient).

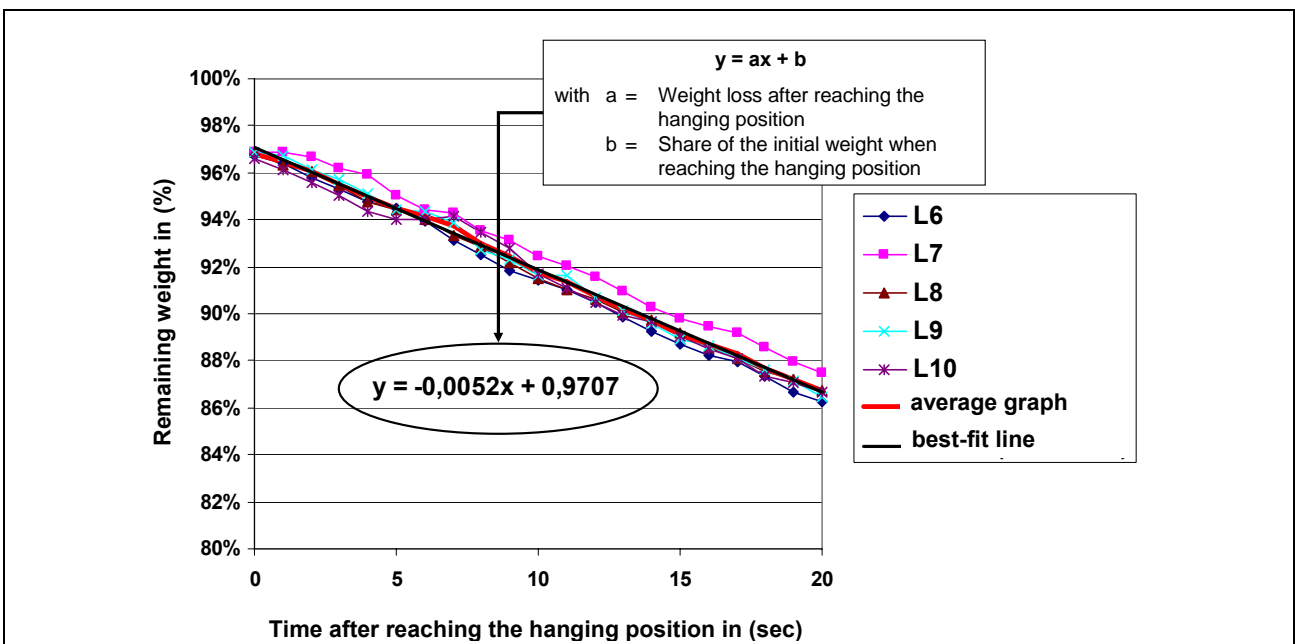


Figure 10: Evaluation diagram concerning weight change [5]



The example shows pellets from material B under use of lime as a binding agent after a drying time of 18 hours and at a temperature of 950 °C an average a decrease in weight of 2.93% during the manual hanging of the pellet into the furnace. During the next 20 seconds in the hanging position the pellets lose an average mass of 0.52 % per second.

This procedure was done for all types of pellets for different temperature steps. From the data values results findings about the behaviour of the pellets during the hanging procedure until reaching the hanging position in the furnaces as well as after reaching the hanging position.

Parallel to the investigations with regard to the weight change also information about the thermo-shock stability of the pellets were gathered. By the continuous recording of the measuring data it was possible to assign the exact time of bursting for the pellets which did not stand firm of the heating for the whole measuring time. For the assessment of the thermo-shock stability the time scheme shown in table 2 was defined which should come close to the circumstances of a real process in a bath melting reactor of the type ISA-Smelter.

Table 2: Assessment criteria of the pellet bursting depending on the time

Time	Bursting of the pellets during the laboratory trials	Assumption of the bursting of the pellets in the real process
$t \leq 0$ s	The pellet is bursted during the hanging procedure before reaching the hanging position.	The pellet is bursted shortly after charging at the charging opening of the furnace. De-mixing and a high risk of material loss through the off-gas stream is given.
0 s < $t \leq 4$ s	The pellet is bursted shortly after reaching the hanging position.	The pellet is bursted near the melted foamy slag. De-mixing respectively material losses can not be impossible.
$t \geq 5$ s	The pellet is bursted long time after reaching the hanging position.	The pellet is bursted earliest at hitting the melted slag layer. De-mixing with low risk of material losses.

3.2 Test facilities for spontaneous slag contact trials

For the slag contact trials a crucible furnace with a usable crucible volume of five litres was used. The maximum reachable furnace temperature was up to 1,500 °C. In the following picture the cross section of the used furnace is shown.

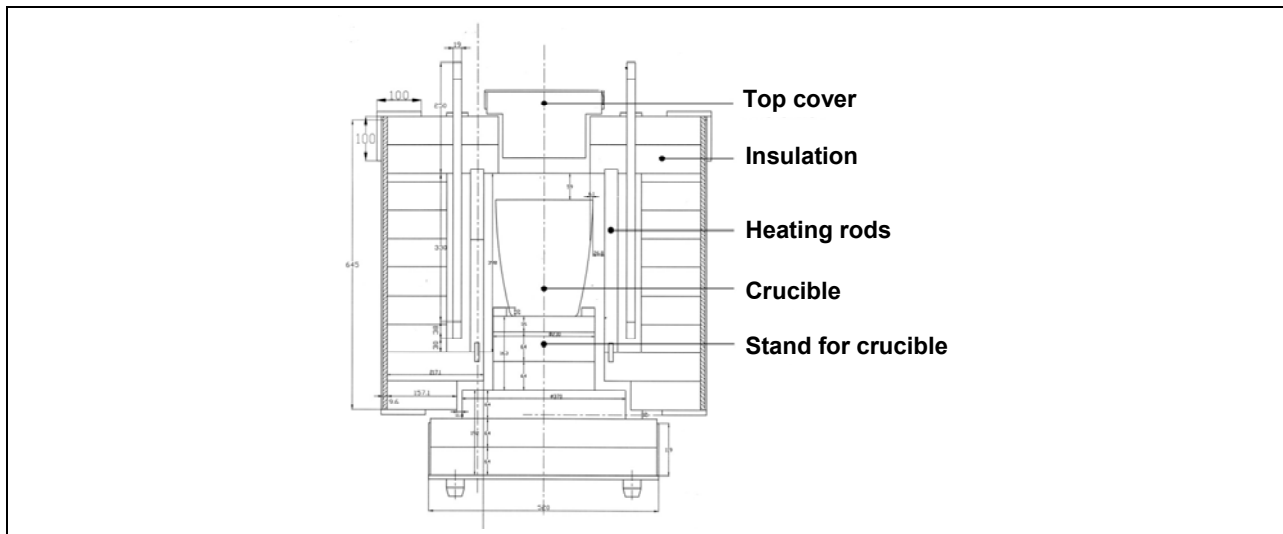


Figure 11: Cross section of the crucible furnace [5]

To realise the free fall of single pellets from a defined height into the moved melted slag as well as the removal of these pellets after defined time intervals, a special equipment was constructed. With this equipment it was also possible to realize the movement in the bath.

A tube from highly heat-resistant Thermax steel was used for the realisation of the defined drop height as a throw-in pipe of the pellets in whose bottom a basket from NiCr-Ni-wire was installed. The Thermax pipe could be moved by means of a pneumatic unit vertically. In the lowest position of the pipe, the test position, the basket installed under the pipe was in the liquid slag phase and acts as a sieve. The pellet which falls onto the melt could be taken out from the crucible by upward movement of the pipe. To create the bath movement the Thermax pipe was moved, in addition, by a rotation engine into a rotary movement, so that the basket located in the melt acted at the same time as a mixing mechanism.

3.2.1 Test parameters and evaluation

For the experimental work a synthetic slag was used, because in the essentials the heat and less the chemistry of the slag bath is used to speed up the reactions inside the pellets. The viscosity of the synthetic slag had to be chosen in such a way that during the removal of the pellet basket from the 1,250 °C hot melt the dripping off of the slag is guaranteed and maximum only a thin slag layer remains around the pellet. The composition of the synthetic slag was of 45 % SiO₂, 42 % CaO and 13g % Al₂O₃. For the trials 1,000 g slag was melted. After addition of 20 g of flux material this slag had at 1,200 to 1,250 °C an enough low viscosity to guarantee the removal of the pellets from the furnace and afterwards from the basket.

Pre-tests shows that the pellets were not changed in their form and size due to the influence of the hot temperature. Although they could be removed easily from the furnace their mechanical stability was strongly reduced. Lightest touches, for instance by a tweezers for taking the pellets out of the basket, led the pellets crumble. For this reason metallographic investigations were not possible. The



assessment of this experimental step was done only by the metallization grade of the removed pellets depending of their residence time on the liquid slag.

4. Results of the experimental works

4.1 Bursting and weight change of the pellets due to the spontaneous heating

With long as well as short drying time a high share of clearly more than 90% of the investigated lime-bonded as well as cement-bonded pellets are resistant concerning a spontaneous heating. With a drying time of 24 hours the bursting of the pellets increase, whereas more of 60 % of the pellets from the coarser materials A and B remain stable. The bursting of this pellets take place after longer residence time in the furnace. The finer material C shows clear differences between the both used binding agents. The lime-bonded pellets from material C have compared to the cement-bonded pellets nearly no stability concerning the spontaneous heating. Probably due to the low water addition during the pelletizing of the fine material not enough lime hydrate will be generated, which leads after hardening to the formation of stable solid state bridges.

A dependence of the bursting on the furnace temperature was not recognizable in the experimental works.

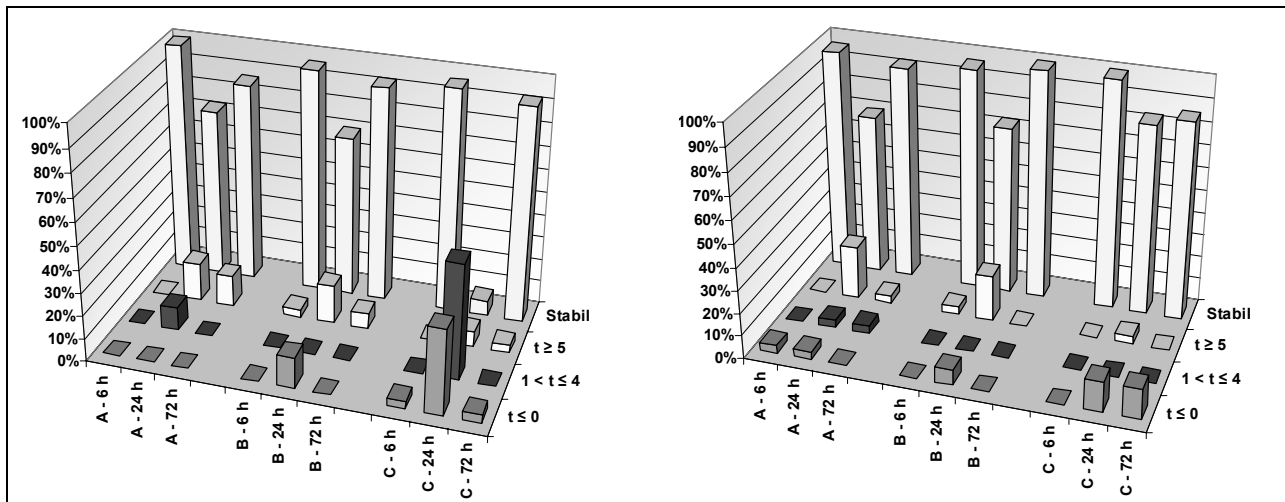


Figure 12: Share of bursted pellets depending on the residence time in the furnace (left: Binding agent lime; right: Binding agent cement) [5]

Concerning the weight change the six investigated types of pellets differ in their behaviour partially only slightly. In particular pellets from material A and material B are very similar to themselves. The mass change up to the reaching of the hanging position in the furnace amounts with pellets from material A and B under use of lime as a binding agent to maximum 4 % or to 5 % or in both cases by use of cement as a binding agent 7%. The decrease in weight accumulated about all tem-

perature steps amounts as a function of the drying time between 2.5 to 3.8 % per second. As a rule the decrease in weight is lower with longer drying time of the pellets and rises with decreasing drying time.

The behaviour of pellets from material C against it does not correlate in such a rate with the described behaviour of pellets from material A and B. Weight losses up to the reaching as well as after reaching of the hanging position are clearly low as with the Pellets from other both materials. Nevertheless, they show no typical behaviour as a function of the drying time.

The amount of liquid for the production of the pellets is depending on the mean grain size of the used material. This arises in the clearly lower mass losses of the Pellets from material C. It is interesting concerning a systematic behaviour that pellets from material B, which has the highest mean grain size, have clear characteristics depending on the drying time. Due to the coarse basic material they probably have a relatively coarse grain structure with bigger ones or more open pores, than Pellets from the finer basic materials. This assumption can be confirmed by the behaviour of pellets from material A which is slightly finer than material B. They behave as Pellets from material B, but not in this clearness. However, pellets from material C, which is significantly finer, cannot be compared concerning her behaviour to the other types of pellets.

4.2 Geometrical change with spontaneous heating

The visual evaluation shows, like in the test results for the weight change, clear differences in the behaviour of the pellets depending on the mean grain size of the used materials.

The following picture shows exemplarily a degassing process, which was observed by the camera system.

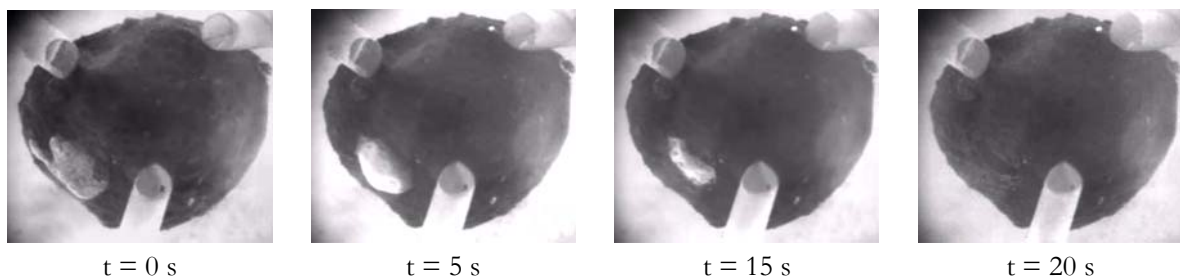


Figure 13: Evaporation effect on the pellet surface (lower left side) [5]

The pellets from material A and B show mainly a comparable behaviour. In particular this becomes clear with short drying times. Degassing processes in combination with drop formation at low and middle test temperatures predominate. At high temperatures from 1,050 °C these effects are removed by spalling effects. Pellets from material C show this behaviour also, but not with lime as a binding agent, but with cement. With cement-bonded pellets from material A and B light trends towards spalling effects are to be noted with short drying time partially with gasification effects.



It is confirmed, that for 24-hour drying time the bursting of the pellets is relatively high for all types of pellets. A systematic occurrence of the described effects cannot be seen. The following diagram shows exemplarily the effects for one of the investigated pellet types.

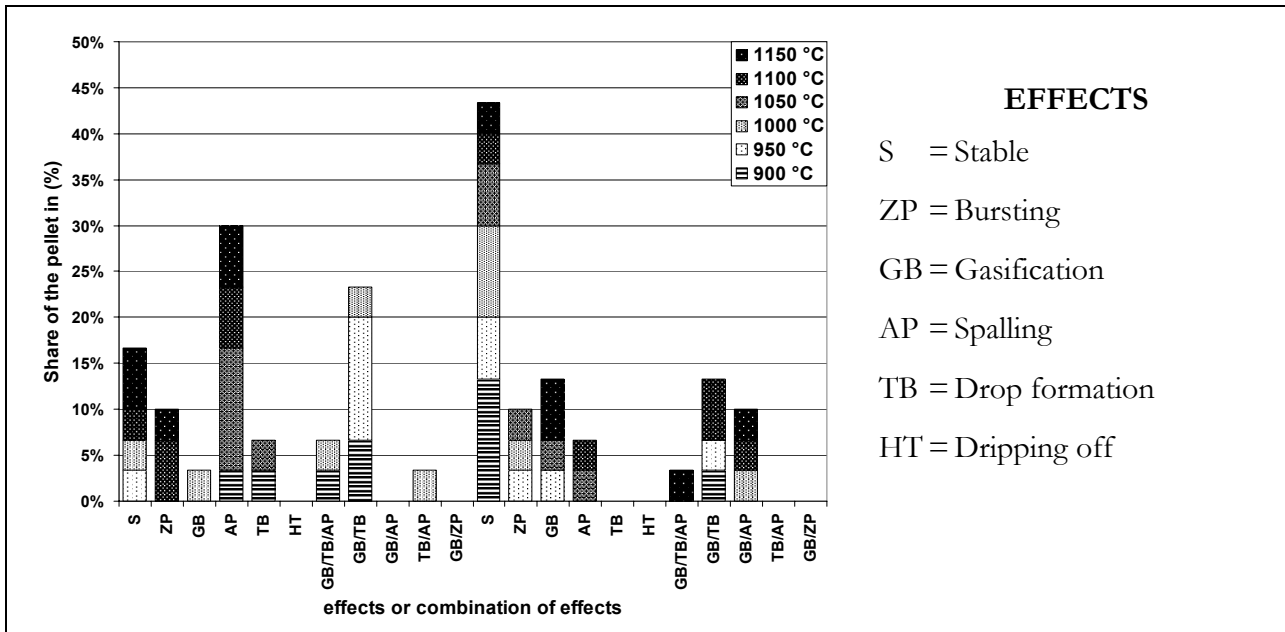


Figure 14: Effects or combination of effects of pellets from material B at $t_d = 6$ h depending on the temperature (left: lime-bonded; right: cement-bonded) [5]

The different behaviour of lime- and cement-bonded pellets can be explained by the hardening behaviour of the binding agents. The binding power of lime is based on the formation of lime hydrate by the moisture absorption during pelletizing. This is the base for the hardening, because a slow absorption of carbon dioxide from the ambient air under formation of carbonic acid and reaction with the formed lime hydrate to calcium carbonate as a solid state bridge connection takes place. Because the process of the carbonation is protracted, it can be assumed that this kind of the solid state bridge formation takes not effect with the pellets, but rather the liquid bridges rule: This also explains why the drier lime-bonded pellets tend to an increased bursting.

The hardening of cement as a binding agent is compared to lime based on the quick formation of calcium silicate hydrate as well as lime hydrate crystals. These form a firm structure between the single particles of the pellets. This is possibly the reason, why with cement-bonded pellets spalling effects dominate.

In total it is shown that pellets from all three basic materials are suitable for the spontaneous heating. From economic view and also due to the described experimental results a short drying time is advantageous. On the one hand storage spaces are thereby decreased in the production plant, on the other hand, the bursting effects are very low. Lime-bonded pellets with short drying time tend to spalling effects only at high temperatures. Compared to the charging procedure of a real process in a bath-melting reactor the pellets are in this case near of the liquid foamy melt, far away from the



suction effect of the exhaust gas system of the furnace. For this case loss of material is less expected.

4.3 Metallization grade of the pellets after slag contact

The metallization grade equals to the mass share of the respective element in metallic form in the total mass of the analysed pellet. The metallization grade should allow an inference on reduction processes in the pellets. It is expected that the share of metallic copper and iron increase and the share of volatile zinc decreases. As the following picture shows, these expectations are not fulfilled. Except the pellets from material A the reduction of the metallic shares is to be registered with all investigated materials by trend, as shown in the following diagram.

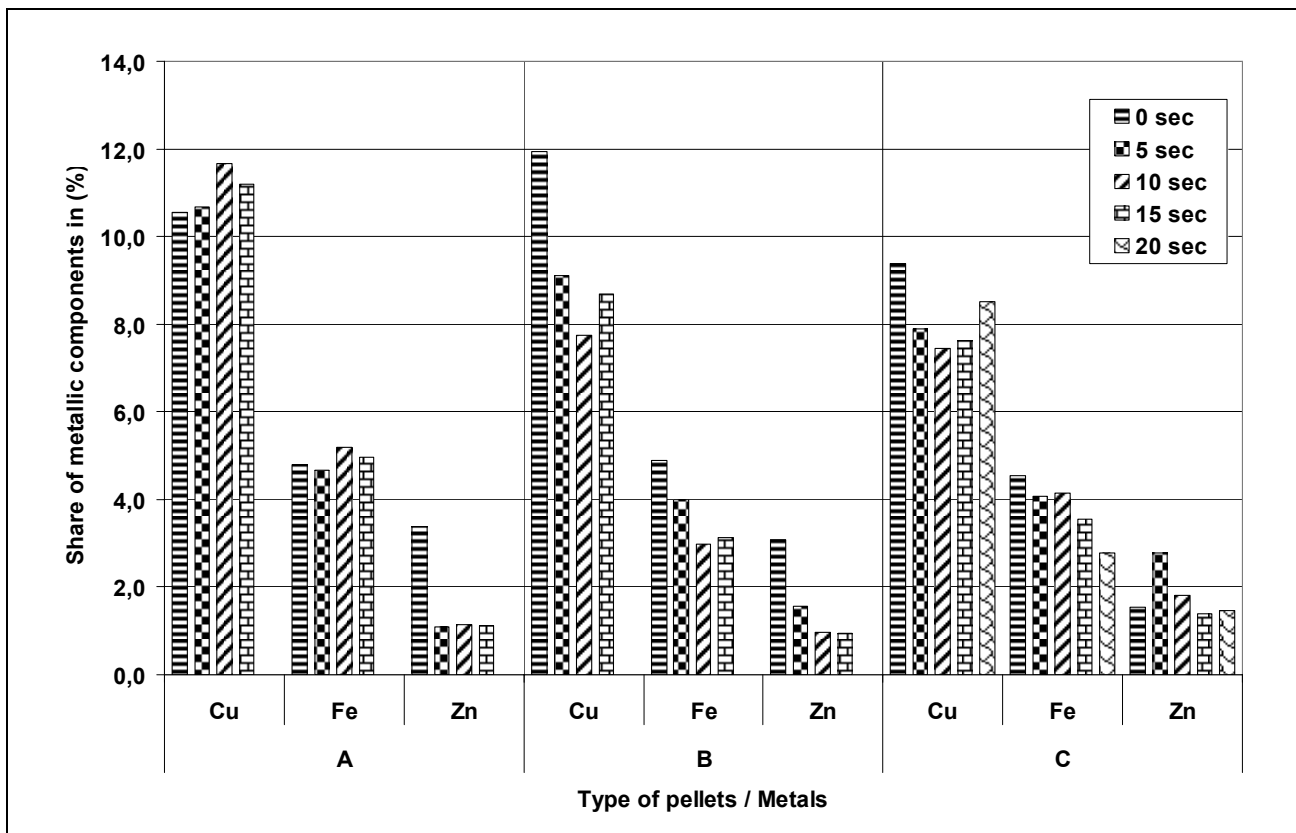


Figure 15: Average grade of metallization of the different types of pellets depending on the residence time on the slag [5]

The results are to be explained mainly by the fact, that the used furnace system not allows the realisation of an inert furnace atmosphere. The contact with aerial oxygen causes probably oxidation processes in the pellets. This happens a little already during the fall through the Thermax pipe, but mainly if the pellet swims on the slag melt, because it is from above in contact with the non-inert atmosphere. But even if an inert furnace atmosphere had been realisable, the pellet comes after removal certainly in contact with aerial oxygen. Nevertheless, the metallization grade of the zinc seems to be realistic. Zinc is a volatile metal, irrespective of the furnace atmosphere from 1,000 °C



evaporation takes place. Beside the metallic zinc already contained in the pellets, oxidic zinc compounds are reduced at the used temperatures in interaction with the pyrolysis of organic contents to gaseous zinc.

Beside the influence of aerial oxygen the metallic components in the basic materials can also hinder the evaluation of the results. In particular metallic copper and iron components can vary from pellet to pellet extremely. Wires, which are integrated in the pellet structure, are also to have be mentioned. Copper wires can start to melt at the surface of the pellets at a furnace temperature of 1,250 °C with increasing residence time, drip off into the liquid slag layer and therefore also influence the metallization grade of the taken out pellets.

5. Assessment and Outlook

The first experimental step shows that pellets from all three basic materials are suitable for the spontaneous heating. From economic view and also due to the described experimental results a short drying time is advantageous. On the one hand storage spaces are thereby decreased in the production plant, on the other hand, the bursting effects are very low. Lime-bonded pellets with short drying time tend to spalling effects only at high temperatures. Compared to the charging procedure of a real process in a bath-melting reactor the pellets are in this case near of the liquid foamy melt, far away from the suction effect of the exhaust gas system of the furnace. For this case loss of material is less expected.

The second experimental step shows that the pellets endure the free fall on the moved liquid slag without damage and remain stable in their form. Pellets from the coarse materials A and B are after maximum 15 seconds of residence time on the slag still removable, pellets from material C even after 20 seconds.

In conclusion it can be said that the pellets from the fine material mixture C under use of lime as binding agent achieve with a low drying time of 6 hours the best results within the scope of this work. The positive aspects are in particular the easier production of the pellets, the low abrasion as well as the behaviour with the spontaneous heating. However, is to be mentioned that the results of the pellets from both coarse material mixtures A and B, also lime-bonded and with low drying time, are not to be assessed much worse.

For the transfer of the results on the real conditions of a copper plant other boundary conditions must be investigated and analysed more thoroughly. This concerns, for example, the mechanical forces which have an impact on the pellets during pelletizing in industrial scale. Also internal transport processes and storage conditions are to be assessed separately. To produce pellets which are stable enough for these mechanical impacts, binding agent amounts are to be raised if necessary and possibly additional additives must be added. The question stays open if an uncontrolled agglomeration of the material mixtures by enough high water addition, taking into account a raised energy



consumption while melting, is still more economically than a perhaps excessively raised binding agent addition with all logistic and economic expenditures linked with it.

The internal material flow in a plant can be influenced by a successful use of pellets, in spite of the additional logistic expenditure at the beginning of the process in the material preparation, in total positively. The recovery of blister copper and iron silicate sand can be raised by reduced dedusting effects and therefore raised material supply in the reaction area of a bath melting reactor and at the same time disadvantageous effects in the exhaust gas system can be reduced.

6. References

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