Zbyszek SZELIGA*, Bohumír ČECH**, Jan MATOUŠEK***, F. WINTER****

THE POTENTIAL OF ALTERNATIVE SORBENTS FOR DESULPHURIZATION – LABORATORY TESTS

LABORATORNÍ TESTY ALTERNATIVNÍCH SORBENTŮ PRO ODSÍROVACÍ PROCES SUCHÉ ADITIVNÍ METODY

Abstract:
At present, the natural limestone is used for desulphurization of waste gases for combustion of fossil fuels. However, it is important to save all primary sources for the future, i.e. this applies for limestone as well as. The researchers focused on potential finding the alternative sorbents for the purpose of desulphurization in dry additive method of desulphurization, which would become the alternative for natural limestone. The article is primarily focused on desulphurization tests of selected substances, i.e. starting from tests in laboratory scale to pilot scale units to the tests in real combustion facilities.

Abstrakt:
Pro odsiřování spalin ze spalovacích procesů je v současnosti převážně používán vápence, primární přírodní surovina. Je bezprochy věrně důležité šetřit primární zdroje surovín, mezi které patří i zdroje vápence. Tento článek se pak zabývá výzkumem autory zadefinovaných náhradních sorbentů pro suchou aditivní metodu odsiřování. Jsou popsány původní výsledky výzkumu na modelech fluidní vrstvy.

1 Introduction

Alternative sorbents are defined by authors as waste substances, by-products from paper industry production (caustic sludge) and from sugar industry production (saturation sludge), see table no.1, and they represent, under the present conditions of knowledge, the alternative of the so far most frequently used sorbents – natural limestone.

The general motivation for selection of these substances was the fact that these substances are waste materials from the below described processes. These substances may replace the primary natural raw material – limestone - as the secondary raw material.

1.1 A brief description of origin of tested substances:

1.1.1 Caustification sludge from paper mill production:
In principle, the caustification process as an important part in paper mill technology is transformation of sodium carbonate, which is generated in combustion of black precipitated waste liquor, to sodium hydroxide. This occurs with addition of limestone, which is slaked directly in green liquor. This reaction takes place in two stages:

- \[ \text{CaO} + \text{H}_2\text{O} = \text{Ca(OH)}_2 \]
- \[ \text{Ca(OH)}_2 + \text{Na}_2\text{CO}_3 = 2\ \text{NaOH} + \text{CaCO}_3 \]

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Limestone is slaked in green liquor, the suspension is led through the series of causticizers into the sedimentation tanks with white liquor where the sludge is separated. The caustification sludge will be washed in vacuum filter and it will be dosed into the limestone kiln where it is burnt to technical calcium oxide, which returns again to production.

Limestone burning from the caustification sludge is a similar process as limestone production at the limestone works. In principle it is a thermal decomposition of calcium carbonate

$$\text{CaCO}_3 = \text{CaO} + \text{CO}_2$$

In comparison with limestone used at the limestone works, the caustification sludge contains 45 up to 55% of water, which should be evaporated. The rotary furnaces or fluidized reactors are used for regeneration.

Then the caustification sludge is a special waste generated in production of cellulose by sulphate method in the case when it is not returned to the process. In principle, it is the condensed calcium carbonate. At large plants, calcium oxide is regenerated from calcium carbonate by burning in the rotary furnaces and it is used again for preparation of cooking liquor. Even with this technology the return of calcium carbonate is not always continuous and in the case of small-scale and older technologies the caustification sludge is discharged to the waste disposal sites.

As an example, the waste disposal site at the paper mill in Štětí can give an imagination of the quantity and availability of caustification sludge where the quantity of this substance in the lagoon of approx. 5 ha was deposited in the depth up to 10 m during the decade of paper mill production and from where the sludge was extracted for desulphurization experiments.

1.1.2 Saturation sludge from sugar industry

The saturation sludge is produced as by-product in production of sugar from sugar beet. At sugar factories the burnt limestone and carbon dioxide are used for treatment of sugar juice by the so-called evaporation that are produced by each sugar factory in the limestone kiln. The basic raw material is lump saturation limestone, which is burnt at temperature of 1100°C and it is decomposed to carbon dioxide and calcium oxide. The fuel is coke made of the Ostrava hard coal. The consumption of coke is 7-10% of limestone weight. Limestone is burnt in the shaft furnace and it is dosed from the top. The charge falls down by own weight and the burnt limestone is taken off in the lower part of the furnace. The combustion air needed for coke burning is blown into the lower part of the shaft furnace. The produced waste gases (saturation gas) are extracted in the upper part of the shaft furnace. The saturation gas contains 28-32% of carbon monoxide, 1,5-2,5% of oxygen, the residue is nitrogen and small quantity of sulphur dioxide. Depending on the excess combustion air the saturation gas may contain up to 1% of carbon monoxide, however, the production of CO is not desired. Then the saturation gas is cooled down and sprayed, thus dust particles and acidic components in gas are removed, primarily SO₂.

From burnt limestone CaO the limestone solution in concentration of CaO 215 kg·m⁻³, is prepared from pure water or from sweet water and then mechanical contaminants are removed from this solution.

The following processes and reactions take place by effect of limestone, limestone and hydroxide ions in purifying of sugar juice.

- Precipitation of high molecular sugars – pectin, proteins, etc.
- Precipitation of anions in the form of limestone salts (phosphates, sulphates, etc.)
- Precipitation of magnesium ions
- Invert sugar decolourizing with production of lactic acid and formic acid
- Parts of crushed beet are caught in limestone precipitation

Then, in this way produced precipitation is defecated with addition of saturation gas and Ca(OH)₂ is transformed into crystalline CaCO₃, which simultaneously serves as the suitable filtration agent on which surface colour substances and precipitated non-sugar substances are caught.
The above-mentioned processes take place in two stages of pre-defecation and final defecation with subsequent saturation. After saturation the saturation sludge is separated by sedimentation and filtration.

The produced saturation sludge is a significant by-product from sugar production.

From 1 ton of raw sugar beet the 130 kg of sugar is produced in average. According to literature, it is 6-8% of saturation sludge for defecation per one ton of beet, according to the state-of-the-art technique the value is approx. 5,5%.

The existing utilization of saturation sludge is as follows:

- Fertilizer for treatment of acidic reaction of soils, for direct spreading on the field the dry matter shall be about 70%, organic mass in sludge is easily transformed into humus and it is an ideal substrate for microbiological activity, the use of sludge improves soil structure
- Fertilizer for gardeners with additive of approx. 10% of limestone powder
- Mineral additive into fodder mixtures after mixing with pressed cuttings and molasses

At present the saturation sludge is used as the fertilizer for agricultural fields. The sludge is continuously transported during the period of sugar beet campaigns, the sugar factories have not any storage areas. The transport vehicles that arrive with sugar beet in the sugar factory take away the raw sludge.

(The average balance of saturation sludge production in the Czech Republic is approx. 211000t/year.)

The present utilization of saturation sludge is primarily in agriculture as the fertilizer as mentioned above and to a smaller extent it is used as the additive for fodder mixtures for animals. However, the saturation sludge is not utilized 100%. This substance could be more utilized for desulphurization purposes. In addition, the utilization of this substances by sugar factories could be financially beneficial and bring economic improvements for sugar factories.

The high content of CaCO₃ (and/or CaO) and high content of water are striking features of these substances. The other important parameter of these substances identified as alternative sorbents is their granularity and their behaviour during utilization. The grain size analyse for saturation sludge is shown in Fig. 8. Microscope photo see Fig. 9

1.1.3 Limstone VFK 55 and VFK 80

As reference substance to alternative sorbent, caustification sludge and saturation sludge the limstone from the Štramberk location was used and it is identified as VFK 55 and VFK 80. Chemical composition see Table no.2 and grain size of these types of limestone (VFK 55) are shown in Table no.3. These types of limestone, sorbents are used for desulphurization in some fluidized-bed boilers in the Czech Republic. The abbreviation VFK means a trade mark, limestone for fluidized-bed boilers, the following number identifies the parameter of grain size, it informs on the screen oversize of 90 μm.

2 Experimental Results and Discussion

After general research into chemical and physical properties of investigated waste substances suitable for dry additive method of desulphurization in fluidized layer and their study, the authors found out the already mentioned set of substances. These substances are appropriate for the desulphurization process not only from the viewpoint of the above-mentioned properties, but also because of their availability. Their availability may be one of criteria for their real implementation in heavy duty facilities.

Prior to application of alternative sorbents in the process of dry additive method of desulphurization in real equipment, there was necessary to test the behaviour of sorbents in laboratory
conditions. For this purpose, the pilot units at TU-Vienna and at the Czech Academy of Sciences in Prague were chosen.

The composition of pilot tests was selected primarily for the purpose of suitable combination of the gained knowledge.

2.1 Fixed Bed Reactor Tests

Pilot tests started with verification tests of conversion (sorption capability SO₂) of alternative sorbents and conventional limestone used in fluidized technology as reference substances on the equipment with immobile layer RRU, see Fig. 1 at the laboratory of TU-Vienna.

The sorbents mentioned in table 1 and limestone VFK 55 and VFK 80 were used for tests.

The equipment consists of electrically heated reactor, immobile bed and system for time measurement and analysis of outlet gases, primarily the content of SO₂.

During the tests the researchers had to cope with the problem of coagulation of samples, from this reason it was necessary to use siliceous sand in ratio of 1:9 in sample matrix. Then this solution guaranteed approximately the same porosity of layer and thus the same velocity of gas flow through all samples.

The prepared sample, a mixture of the given sorbent of constant weight and siliceous sand in the given ratio was positioned on the grate, uniformly spread over and incrementally heated up to the temperature of 850°C, while N₂ was simultaneously flowing as inert gas. After stabilization of temperature in the reactor, nitrogen was replaced by a mixture of gases SO₂=790ppm; CO₂=15%; O₂=6%; the remainder was nitrogen. Gas concentrations were constant during the whole period of tests. Gas concentrations were monitored on the outlet from the unit during the whole duration of test.

The criterion conversion of SO₂ in the given time interval for all substances was chosen as the parameter for evaluation of results from tests. These results from tests on the RRU equipment are shown in table 4. and Fig. 2.

The parameter of conversion was defined as follows,

\[ \text{conversion} = \frac{A_2}{A_1 + A_2} \times 100 \% \]

\[ A_1 = \int_{\tau_1}^{\tau_2} C_{SO_2} \, d\tau \]

\[ A_2 = \left[ C_{SO_2, \text{max}} \times (\tau_2 - \tau_1) \right] - A_1 \]

From results gained from this test it is apparent that conversion of tested alternative sorbents is greater than of traditional limestone, this may be attributed to granularity of the mentioned substance and thus to their large, specific and reaction surface. This test confirmed assumptions that these substances may be implemented in dry additive method of desulphurization. Then in next tests it is necessary to investigate these substances in real process, i.e. in the fluidization process and with this related time of dwell of the ultra-fine substances in the space of fluidized layer and conditions suitable for desulphurization reaction. Then, according to theoretical assumptions, the efficiency of desulphurization process is most probably the question of ratio between the kinetics of reaction, its velocity and the time of dwell of the particles in the fluidized layer.

2.2 Tests on model of fluidized furnace with circulating layer CFB

After gaining the general information on behaviour of alternative sorbents in the process of dry additive method of desulphurization with stationary layer, the next step for identification of properties of alternative sorbents was made on the model of boiler with circulating fluidized layer. A chart of pilot equipment located at the laboratory of TU Vienna is shown in Fig. 3. The equipment repre-
sents a simplified model of fluidized-bed boiler with circulating layer. The equipment is thermally insulated, the temperature inside the equipment is maintained by electrical heating.

The simulation of flow of waste gases through the chamber is provided with a mixture of gases, the mixture was supplied to the chamber as fluidized medium and its composition was chosen similarly as for the RRU test, the difference in concentration of SO$_2$ are caused by technical constraints, the possibilities of regulation system of gas flow.

The composition of flow of simulated waste gases: the mixture of gases SO$_2$=525ppm; CO$_2$=15%; O$_2$=6%; the remainder was nitrogen.

The composition of gas and temperature 850°C were maintained during tests on the constant value. The siliceous sand was used as bed material.

After stabilization of all general parameters of unit the sorbent was one-off dosed by a dosing equipment into fluidized layer. Then the response was observed as the drop of concentration of SO$_2$ on outlet from the unit. Results from test are illustrated in table 5, the evaluation of result was subjected to the same criterion as in the RRU test, i.e. the evaluation of Conversion of monitored substances in the specified time interval.

The time dependence of concentration SO$_2$ on the outlet from the unit after implementation of sorbent batch is shown in Fig. 4. When comparing the results of Conversion obtained on the RRU and CFBC for the given substances it is clear that the comparable results were obtained on both units. It is very interesting that the Conversion of alternative sorbents is better in the process of dry desulphurization on the model of fluidized layer than in the case of traditional limestone used in the fluidized furnaces.

The time of stay of alternative sorbents in fluidized layers is shown as very interesting and very surprising.

During these tests the time of stay of sorbent particles by means of response of concentration of SO$_2$ and other relevant quantities were observed. According to analyses, see enclosed analyse of grain size of saturation sludge (like example) in. fig. no. 8, these substances are ultra-fine. The time of stay of these substance in fluidized layer during the test does not correspond to calculated assumptions. Then this phenomenon has determined one of research objective of this project.

During the investigation of this phenomenon the authors came to the unambiguous conclusion, hypothesis on the basis of many results and primarily according to the evaluation of microscopic photos of the mentioned substances. The conclusion may be generalised in such a way that the investigated ultra-fine sorbents will stick on particles of bed material of fluidized layer that are then a “carrier” of these grains and they together circulate in the layer.

2.3 Pilot-Scale 100 kW FBC Tests

To approach more to real equipment, for the purpose of testing the measurement was carried out on the equipment at AV ČR ÚCHP.

Tests were carried out on the pilot laboratory unit with rated output of P$_{jm}=100$kW with stationary fluidized layer, see Fig. 5.

Desulphurization tests took place with combustion of mixture of Sokolov brown coal, with individual alternative sorbents with reference substances, natural limestone. The mixtures (coal + individual sorbents) were prepared in molar ratio of Ca/$S=2.4$ for maintenance, simulation of real conditions in fluidized furnaces of real units for all investigated substances.

Then every prepared mixture was supplied before its testing into the bunker no. 1, from which the mixture was dosed by a screw feeder into the combustion chamber of corresponding weight flow. When the reserve of prepared mixture was exhausted, the dosing with clean coal began from the bunker no. 2 to the boiler. In this time interval of coal dosing, a new mixture was prepared and supplied into the bunker no. 1. Then the combustion of coal itself gives the opportunity to obtain data for determination of the level of SO$_2$ without supply of sorbent and primarily there is the possibility to eliminate the influence of test by sorbent from the previous test. As results from tests there are stated
the average concentration of SO₂ in the course of individual tests, and time dependence of concentra-
tion of SO₂ during tests, see Fig.6

From obtained results one may already see the effect of furnace size, the obtained results con-
firm the so far gained results from the laboratory units mentioned above.

In comparison with traditional limestone, the alternative sorbents show very good properties in
the desulphurization process.

3 Conclusions from carried out tests

Within the framework of presented papers there was carried out the general research of sub-
stances that were appropriate for implementation in dry additive method of desulphurization in fluid-
ized layer. The types of substances with appropriate physical and chemical properties and their avail-
ability were defined. According to the determination of general chemical and physical properties the
saturation and caustification sludge was selected as the most suitable substance.

Laboratory desulphurization tests were the basic step of research of these substances and these
tests were carried out on models of combustion units.

The research of behaviour of alternative sorbents in laboratory conditions in dry additive
method of desulphurization prepared the base for performance of tests on real equipment. Results
from tests may be evaluated as very good with the prerequisite for utilization, testing of investigated
substances in real combustion units.

On the basis of carried out laboratory and pilots tests one may expect good results from these
real units (equipment with greater output), some of these experiments have been already performed.

Fig.7 illustrates results of research of alternative sorbents on individual models of combustion
units. It may be stated that results of the given sorbents on the given equipment are in mutual accord.
Primarily by comparison of results from the unit CFBC and unit FBC \( P_{\text{m}}=100\text{kW} \) it may be con-
cluded that the tests on the model CFBC were carried out correctly and the designed unit is very satis-
factory. Some aspects of application of ultra-fine sorbents will be further investigated on this unit.

The process of stay of ultra-fine particles of alternative sorbents in fluidized layer of model
CFBC was recorded as one of phenomena of behaviour of investigated substances for the period ex-
ceeding the period calculated on models. Further, the hypothesis was defined and partially examined,
which explains this phenomenon.

Authors in this article attempted to describe the research of alternative sorbents that have a
great chance to replace the primarily natural raw material, limestone. It should be emphasized that
waste substances were identified and investigated as alternative sorbents. This article describes indi-
vidual steps of research, primarily laboratory tests on the real unit.

It was carried test with the saturation sludge in unit with circulating fluidized layer with rated
output of \( P_{\text{m}}=120\text{MW} \), with the saturation sludge as the substitute for natural limestone the results of
laboratory tested were confirmed.

The desulphurization test was also carried out with the saturation sludge in the powder boiler
with granular furnace \( P_{\text{m}}=72\text{MW} \), the results appear already as very good.

The preliminary conclusions from these tests confirm a very good sorption capability on sub-
stances identified by authors as alternative sorbents in the process of dry additive method of desul-
phurization of the given technologies.

However, it is necessary to carry out other tests and primarily tests of long-term character.

*The article was prepared with support of GAČR 101/03/D187*
### Table 1 Chemical analysis of alternative sorbents

<table>
<thead>
<tr>
<th>Sample</th>
<th>SiO₂</th>
<th>Al₂O₃</th>
<th>FeO</th>
<th>Fe₂O₃</th>
<th>CaO</th>
<th>Na₂O</th>
<th>H₂O</th>
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<td>Saturation sludge A, sugar factory</td>
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<td>0,31</td>
<td>51,37</td>
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<td>Caustic sludge C, Chomutov</td>
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<td>53,91</td>
<td>0,310</td>
<td>43,39</td>
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### Table 2 Chemical analysis of VFK 55 and VFK 80

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<th>Al₂O₃</th>
<th>CaO</th>
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### Table 3 Grain size of VFK 55

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### Table 4 Results of conversion in RRU

<table>
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<th>Tested substance</th>
<th>Conversion [%]</th>
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<td>Saturation sludge A</td>
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<tr>
<td>Caustic sludge (Štětí) B</td>
<td>51</td>
</tr>
<tr>
<td>Caustic sludge (Chomutov) C</td>
<td>50</td>
</tr>
<tr>
<td>VFK 55</td>
<td>38</td>
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<tr>
<td>VFK 80</td>
<td>32</td>
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<tr>
<td>Ca(OH)₂</td>
<td>53,3</td>
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### Table 5 Results of conversion in CFBC

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<th>Substance</th>
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<td>Saturation sludge A</td>
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<td>Caustic sludge (Štětí) B</td>
<td>22,5</td>
</tr>
<tr>
<td>Caustic sludge (Chomutov) C</td>
<td>18,4</td>
</tr>
<tr>
<td>VFK 55</td>
<td>14,3</td>
</tr>
<tr>
<td>VFK 80</td>
<td>14,1</td>
</tr>
</tbody>
</table>
Table 6  Summary of FBC tests ($P_{in}=100$ kW)

<table>
<thead>
<tr>
<th>Test no.</th>
<th>Mixture of</th>
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<tr>
<td>Test no. 1</td>
<td>coal + caustic sludge B (from location Štětí)</td>
</tr>
<tr>
<td>Test no. 2</td>
<td>coal + caustic sludge C (from location Most)</td>
</tr>
<tr>
<td>Test no. 3</td>
<td>coal + calcium hydrate Ca(OH)$_2$</td>
</tr>
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<td>Test no. 4</td>
<td>coal + limestone VFK 55</td>
</tr>
<tr>
<td>Test no. 5</td>
<td>coal + saturation sludge A (from sugar factory)</td>
</tr>
</tbody>
</table>

Fig. 1 Chart of RRU equipment

Gas mixture

SO$_2$...790 ppm
CO$_2$...15%
O$_2$ ... 6%
N$_2$ ... remainder

Tested substance

frit

Electrical heating of reactor

TC1, TC2... thermocouples for temperature regulation in reactor

SO$_2$ = f(τ)
Fig. 2 Time behavior of SO$_2$ on the outlet from RRU Unit

![Graph showing the concentration of SO$_2$ over time with annotations for VFK 55, VFK 80, Caustic sludge C, Saturation sludge A, and Caustic sludge B.]

Fig. 3 Model of fluidized furnace with circulating layer, TU-Vienna

![Diagram of a fluidized furnace model with labels for Ash blower, To chimney, To gas analysis, Heating shell, Fuel feeding, Quartz glass, Quartz glass tee with 2100mm height & D 35mm, Heating bandage, Gas mixture, N2 and air, Ash collection, P, T1, T2, T4, T6, C.]

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Fig. 4  Time behaviour of concentration of SO2 on the outlet from boiler model with circulating fluidised layer CFBC after dosing the sorbent.

![Graph showing concentration of SO2 over time]

Fig. 5  Fluidized-bed boiler with output of $P_{jw}$ 100 kW and furnace dimensions 300 x 300mm at the Institution of Chemical Processes of Czech Academy of Sciences.

![Diagram of fluidized-bed boiler]

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Summary of tests, Fig. 6:

<table>
<thead>
<tr>
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<td>coal + calcium hydrate Ca(OH)₂</td>
</tr>
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<td>Test no. 4</td>
<td>coal + limestone VFK 55</td>
</tr>
<tr>
<td>Test no. 5</td>
<td>coal + saturation sludge A (from sugar factory)</td>
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Fig. 6 Time behaviour of concentration of SO₂ in waste gases during combustion tests on boiler 100 kW UCHP ČAV with alternative sorbents and limstons
Fig. 7 Results of sorption in RRU, CFBC and 100 kW FBC

Fig. 8 Granular analysis of A (saturation sludge)

Fig. 9 Saturation sludge A
4 References


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Recenzent: prof. Ing. Pavel Kolat, DrSc.