

SUOMEN SOODAKATTILAYHDISTYS FINNISH RECOVERY BOILER COMMITTEE

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Understanding Low Temperature Corrosion in Black Liquor Combustion

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Understanding Low Temperature Corrosion in Black Liquor Combustion

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1. Summary and Conclusions

A laboratory set-up to study low temperature corrosion was built and tested. The set-up worked as planned and stable water vapor concentrations were maintained. Corrosion of carbon steel coupons, measured as weight change, with either Na₂SO₄ or NaHSO₄ salt placed on top of the coupon was studied at the temperatures 90, 100, 110 and 120°C, Figure 1. The water vapor concentration was 60 vol% in the experiments, simulating the localized high humidity that can be seen near soot blowers. For the NaHSO₄ covered samples, pitting was found as well as an iron sulfate corrosion product, which is consistent with acid bisulfate solution corrosion. Corrosion increased with decreasing temperatures, with deeper pits at the lowest temperatures. For the Na₂SO₄ experiments, iron oxide formation was observed at 90°C and to a lesser extent at 100°C. No corrosion was observed at 110°C and 120°C under Na₂SO₄.

Hygroscopic salts will absorb water above the water dew point. At a given temperature, the humidity at which a salt absorbs water is called the humidity of deliquescence. Based on the data available from literature, at 60 vol-% water the temperature at which the salts were expected to absorb water was 91 °C for Na₂SO₄ and 106 °C for NaHSO₄. The corrosion seen is consistent with absorption of water by the salts, but this is occurring at a slightly higher temperature than would be expected based on predictions for the humidity of deliquescence based on the literature. Further research is needed both to map the conditions under which low temperature corrosion can occur and to measure the humidity of deliquescence for salts and salt mixtures at conditions relevant to recovery boilers.



Figure 1. Calculated annual corrosion layer thickness based on mass loss after washing off corrosion layer in 4h and 24h tests.

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1. Background

Historically, some low temperature corrosion has been observed in recovery boilers. However, the underlying cause has not been clearly documented. One hypothesis had been the presence of a sulfuric acid dew point. This was the driving force for maintaining an elevated flue gas temperature. For companies to drop their flue gas temperatures lower and consider changing the construction materials of flue gas stacks, it is important to understand the cause of low temperature corrosion and map the conditions in which is can occur.

Corrosion probe studies and SO₃/H₂SO₄ measurements have shown that there is no H₂SO₄ and thus no acid dew point in modern recovery boilers. Furthermore, the measurements at Heinola, where there was high SO_2 , indicate that even in a high SO_2 environment, there is no acid dew point. However, at Heinola, we did see an elevated dew point that was above the water dew point, but well below the acid dew point. The fly ash in recovery boilers contains hygroscopic salts, such as Na₂CO₃, Na₂SO₄, and NaHSO4 etc. These salts may have an impact on the corrosion of the heat exchangers in the flue gas duct. Hygroscopic salts will absorb moisture from the flue gas at a temperature higher than the pure water dew point. When the humidity in the flue gas reaches a critical level the solid salt particle transforms into an aqueous solution, which would lead to corrosion of the heat exchanger. Different salts have their own specific relative humidity at which the particle starts to absorb moisture. This is referred to as the relative humidity of deliquescence. A literature review found that salts can absorb water above the water dew point and that a salt such as NaHSO₄ can absorb water 10-20 °C above the water dew point depending on the relative humidity in the gas. Thus, one leading hypothesis is that if hygroscopic salts are present, water can be absorbed during periods of high humidity and form an aqueous solution at the steel surface, resulting in corrosion.

This initial study is focused around setting up and testing a reactor system for these low temperature corrosion studies and to make the first runs with NaHSO₄ and Na₂SO₄ salts and varying reactor temperature (90-120°C).

2. Experimental

The first part of this study was to set up a reactor system with stable temperature and water vapor concentration. Maintaining stable conditions throughout the experiment is crucial when studying low temperature corrosion due to water uptake by salts. Fluctuations in the water vapor concentration or temperature could lead to false interpretation of the results.

A schematic picture of the final experimental setup is shown in Figure 1. To create a constant feed of water vapor, the water is pumped with a Masterflex L/S pump at a constant flow to the steam generator. The steam generator consists of a thin pipe with heating. The temperature in the steam generator was set to 165° C in order to insure that water was vaporized at a constant rate. The hot steam was mixed with N₂, O₂ and CO₂, and the temperature of the wet gas

mixture flowed to a temperature regulator in which the temperature of the wet gas mixtures was cooled to the desired temperature. After the temperature regulator unit the wet gases flowed through a preheater before entering the quartz reactor, where the corrosion samples were placed, located in a tube furnace. The back end of the quartz reactor was heated to minimize water condensation and water from flowing back into the reactor. The wet gases exited the tube furnace to a water condenser unit where water was condensed and finally dry gases were vented out. With this setup the gas composition, gas flow, water vapor content and temperature can be easily adjusted.



Figure 1. Schematic picture of the experimental setup.

The experimental plan for this work is shown in Table I. The corrosion of carbon steel (ST45) coupons was measured. The gas composition used during the experiments was 60% H₂O, 31% N₂, 7% CO₂, and 2% O₂ and the flow of wet gases was 2 Nl/min. This represents a gas composition near the soot blowers, i.e. a flue gas that is diluted with water vapor. The salts studied were sodium sulfate (Na₂SO₄) and sodium bisulfate (NaHSO₄) and the exposure time was 4 hours with some runs 24 hours to validate the 4 hour test results. The temperatures used during the experiments were between 90, 100, 110 and 120°C.

| Run | Salt | Temperature | Water vapor content | Time | Steel |
|-----|---------------------------------|-------------|------------------------|------|--------------|
| | | (°C) | (%) | (h) | |
| 1 | NaHSO ₄ | 90 | 60 | 4 | Carbon Steel |
| 2 | NaHSO ₄ | 100 | 60 | 4 | Carbon Steel |
| 3 | NaHSO ₄ | 110 | 60 | 4 | Carbon Steel |
| 4 | NaHSO ₄ | 120 | 60 | 4 | Carbon Steel |
| 5 | Na ₂ SO ₄ | 90 | 60 | 4 | Carbon Steel |
| 6 | Na ₂ SO ₄ | 90 | 60 | 24 | Carbon Steel |
| 7 | Na ₂ SO ₄ | 100 | 60 | 4 | Carbon Steel |
| 8 | Na ₂ SO ₄ | 100 | 60 | 24 | Carbon Steel |
| 9 | Na ₂ SO ₄ | 110 | 60 | 24 | Carbon Steel |
| 10 | Na ₂ SO ₄ | 120 | 60 | 4 | Carbon Steel |

Table I. Experimental plan

The corrosion samples were 2x2 cm coupons of carbon steel and between 3-4 coupons were used during one experiment. The coupons were weighed before salt addition and roughly 0.1 g of the studied salt was added on top of the coupon. The carbon steel coupons with salts on top placed on the sample tray are shown in Figure 2. A thermocouple is located at the tip of the sample tray to record the temperature close to the samples during the experiments.



Figure 2. Carbon steel coupons with salt in top placed on the sample tray.

After the experiment one coupon was molded in epoxy and the cross section of the coupon was analyzed with scanning electron microscope (SEM). The remaining 2-3 coupons were washed with ultrapure water to remove the salt residue and then dried with acetone. The cleaned coupons were photographed and weighed in order to determine the weight change. The surface of one cleaned coupon was also analyzed with SEM.

3. Results 3.1. Experimental setup

The water pump was calibrated to obtain the desired water flow to the steam generator, and thus the desired vapor concentration during the experiments. After the experiments the weight difference in the water container was determined in order to calculate the exact water vapor concentration during the experiment. The target gas composition, gas composition with the measured highest water vapor concentration, lowest water vapor concentration and the average gas composition of all of the experiments are shown in Table II. From Table II it can be seen that with the experimental setup the water vapor concentration was within 1.3% of the desired water vapor concentration.

| | Target | Average | Highest | Lowest |
|------------------|---------|---------|---------|---------|
| | (vol %) | (vol%) | (vol %) | (vol %) |
| H ₂ O | 60 | 59.3 | 59.9 | 58.7 |
| N ₂ | 31 | 31.6 | 31.1 | 32 |
| CO ₂ | 7 | 7.1 | 7 | 7.2 |
| O ₂ | 2 | 2 | 2 | 2.1 |

Table II. Target gas composition and gas composition at the measured highest, lowest, and average water flows during the experiments.

The recorded temperatures at the sample tray for the 4 hour NaHSO₄ experiments are shown in Figure 3, 4 hour Na₂SO₄ experiments in Figure 4, and the 24 hour Na₂SO₄ experiments in Figure 5. When the experiments started it took roughly 30 minutes for the temperature inside the tube furnace to stabilize. This is because the steam exits the generator at 165 C and the other temperature controllers have to adjust to get the system temperature down to the target temperature. However, the key is that the temperature is only very slightly above the target temperature for those approximately 30 minutes and won't affect the corrosion results. The temperature was stable during the experiments and the tube furnace was suitable for running experiments in low temperatures. For some reason during the 24 hour 90°C Na₂SO₄ experiment every 4 hours a small decrease in temperature was detected. The reason behind this regular decrease remained unknown.

Based on the observations during the experiments and the measured temperature and water vapor concentration, the experimental setup worked well. The temperature was stable during the experiments and the desired water vapor content was obtained with the experimental setup.



Figure 3. Temperature during 4 hour NaHSO₄ experiments.



Figure 4. Temperature during 4 hour Na₂SO₄ experiments.



Figure 5. Temperature during 24 hour Na₂SO₄ experiments.

3.2. Corrosion results

3.2.1. Visual inspection

A photograph of the carbon steel coupons after the experiments before washing is shown in Figure 6. Sodium bisulfate was extremely hygroscopic, and absorbed moisture from the wet gas flow in all conditions. This can be seen as there was little to no salt remaining on the coupon after the experiments. This was not expected. Based on literature data for the humidity of deliquescence, it was expected that the temperature at which water would no longer be absorbed was _____ °C. Sodium sulfate is much less hygroscopic, and the salt was only slightly moist at the lower temperatures. It can be seen that the salt cake for the Na₂SO₄ experiments are more intact.



Figure 6. Corrosion samples after the experiments.

Figure 7 shows the carbon steel coupons after the salt residue was washed off. After the visual inspection it was clear that NaHSO₄ caused corrosion of the carbon steel at all temperatures with the corrosion being more severe the lower the temperature. This is due to the formation of an acidic bisulfate solution on the coupon. In the experiments with Na₂SO₄, no corrosion was visible after the 110°C and 120°C. After the 4 h experiments at 90°C and 100°C small spots of corrosion could be observed. To these results, 24h runs were made. These runs showed the same, that corrosion occurs at 90°C and 100°C, with more corrosion at 90°C than 100 °C. The corrosion experiment with Na2SO4 at 110 °C was run for 24 hours since if corrosion were to occur at 4 hours it would have been very slight. The coupon from the 24 hour run at 110 °C, however, showed no corrosion.





3.2.2. Weight change

After the experiment the carbon steel coupons were washed to remove the salt residue and then weighed in order to determine the weight change due to corrosion. Generally, the corrosion layer is removed during the washing process and the weight loss is due to the removal of the corrosion product. This weight was compared to the initial coupon weight, Figure 8. The results clearly show that the corrosion was more severe for the NaHSO₄ covered carbon steel samples at lower temperature than at higher temperatures. The 4 h 90°C Na₂SO₄ experiment showed a slight increase in weight after the experiments because the iron oxide was not removed by the washing process. The coupon from the 24 h 90°C Na₂SO₄ experiment, in which the corrosion was more severe, showed a decrease in weight. The formation of iron oxide during the 100°C Na₂SO₄ experiments was so low that no weight change was recorded. At higher temperatures no corrosion product was formed with Na₂SO₄.



Figure 8. Material loss of the carbon steel samples due to corrosion.

3.2.3. SEM analysis

3.2.3.1. Experiments with NaHSO₄ salt

Figure shows the cross section SEM image of the carbon steel covered with NaHSO₄ after the 4h exposure at 90°C. The white flakes are pieces of metal originating from the cutting and polishing of the cross section sample. The dark grey areas are the epoxy mold and the light grey is either salt or salt mixed with the corrosion product. From the SEM image clear pitting corrosion can be observed. This is consistent with acidic corrosion due to the acid bisulfate solution produced when NaHSO₄ absorbed water from the gas. The corrosion products consisted of a mixture of iron, sodium, sulfur and oxygen (analysis spots 1 and 2 on the figure). The spot analysis (spot 3) showed clearly that FeSO₄ is formed as a corrosion product.



Figure 9. Cross section SEM image of the NaHSO₄ covered carbon steel after 4 h and 90°C experiment.

Figure clearly shows the pits formed from the acid corrosion after exposure with NaHSO₄ for 4h and 100°C. The pits are about 10 μ m deep and are filled with FeSO₄. Increasingly deep pits were observed with decreasing temperature.



Figure 10. Cross section SEM image of the NaHSO₄ covered carbon steel after 4 h and 100°C experiment.

Figure 9 shows the top surface of the washed carbon steel coupons after the experiments with NaHSO₄ at different temperatures. After washing, some corrosion product (FeSO₄) remained on the surface of the carbon steel sample used in the 90°C experiment. The corrosion was more severe at lower temperatures than at higher. At the lowest temperature the whole sample was corroded, while at higher temperatures pit corrosion was only detected on the surface of the carbon steel sample. The cross section SEM images of the NaHSO₄ covered carbon steel samples showed increasingly deep pits at decreasing temperature; for the 90°C case the pits were around 19 μ m deep, for 100°C 10 μ m deep, and for 110°C the pits were approximately 4 μ m deep. The pit depth for the 120°C NaHSO₄ corrosion sample was not measured.



Figure 9. SEM surface image using 200x magnification with secondary electron detector of the washed NaHSO₄ corrosion coupons.

3.2.3.2. Experiments with Na₂SO₄ salt

Figure 10 shows the SEM cross section image of the carbon steel sample covered with Na_2SO_4 after 24h exposure at 90°C. The black in the Figure is the epoxy, lightest grey is the steel and the darkest grey is the salt crystals. There was a clear corrosion layer on top of the carbon steel which consisted of iron oxide (spot 2 in Figure 10) and had an average thickness of roughly 10 µm. Unlike with the NaHSO₄ samples, there is not pitting. This is characteristic of water dew point corrosion.



Figure 10. Cross section SEM image of the Na₂SO4 covered carbon steel after 24 h 90°C experiment.

Figure 11 shows the surface SEM images of the washed carbon steel samples after the 4 hour experiments at different temperatures. At 90 °C, there are clear areas of iron oxide on the surface. At 100 °C, there has been significantly less corrosion of the surface, but iron oxide is still detected. No visible corrosion was seen in the corrosion coupon after the 4 hour experiment at 120° C.

Figure 12 shows the SEM images of the surfaces of the washed carbon steel samples after the 24 hour experiments at different temperatures. The same trend with the carbon steel samples was noted after the 4 h experiments as after the 24h experiments. The severest corrosion was seen at 90 °C, some corrosion at 100 °C and no corrosion at 110 °C.

The fact that corrosion is seen at 100 °C, indicates that more work needs to be done to measure the humidity of deliquesce for sodium sulfate at conditions relevant to the recovery boiler.



Figure 11. SEM surface image using 200x magnification of the washed Na₂SO₄ corrosion coupons after 4 hour exposure.



Figure 12. SEM surface image using 200x magnification of the washed Na₂SO₄ corrosion coupons after 24 hour exposure.

PROCESS CHEMISTRY CENTRE



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Conclusions

- The work showed that the studied boilers did not show any signs of potential to low temperature corrosion caused by the condensation of sulfuric acid
- During start-up with heavy fuel oil there is a risk of condensing sulfuric acid
- An elevated dew point was observed due to hygroscopic salts

Dew point over salts







Water "dew point" over salts



Objectives

- Understand the role of hygroscopic salts on low temperature corrosion for various water vapor concentrations
- Build a setup to study this with controlled conditions and with an even flow of water vapor (60vol%) at 90-120°C

Experimental setup



Experimental setup



Experimental work

- corrosion sample:
 - 2x2 cm carbon steel coupon
 - 3-4 coupons per experiment
- ~0.1 g of salt on the corrosion sample
- Total gas flow 2l/min
- Gas composition during experimental runs: 60% H₂O, 31% N₂, 7% CO₂ & 2% O₂
- Corrosion determined by:
 - Weight loss
 - Visual inspection
 - SEM analysis

Experimental setup



Experimental Plan

| Run | Salt | Temperature (°C) | Water vapor content (%) | Time (h) | Steel |
|-----|---------------------------------|---------------------|-------------------------------|-------------|--------------|
| 1 | NaHSO ₄ | 90 | 60 | 4 | Carbon Steel |
| 2 | NaHSO ₄ | 100 | 60 | 4 | Carbon Steel |
| 3 | NaHSO ₄ | 110 | 60 | 4 | Carbon Steel |
| 4 | NaHSO ₄ | 120 | 60 | 4 | Carbon Steel |
| 5 | Na ₂ SO ₄ | 90 | 60 | 4 | Carbon Steel |
| 6 | Na ₂ SO ₄ | 90 | 60 | 24 | Carbon Steel |
| 7 | Na ₂ SO ₄ | 100 | 60 | 4 | Carbon Steel |
| 8 | Na ₂ SO ₄ | 100 | 60 | 24 | Carbon Steel |
| 9 | Na ₂ SO ₄ | 110 | 60 | 24 | Carbon Steel |
| 10 | Na ₂ SO ₄ | 120 | 60 | 4 | Carbon Steel |

(from the experimental setup)

Calculated gas composition

| | Target | Average | Highest | Lowest |
|------------------|---------|---------|---------|---------|
| | (vol %) | (vol%) | (vol %) | (vol %) |
| H ₂ O | 60 | 59.3 | 59.9 | 58.7 |
| N ₂ | 31 | 31.6 | 31.1 | 32 |
| CO ₂ | 7 | 7.1 | 7 | 7.2 |
| O ₂ | 2 | 2 | 2 | 2.1 |

Temparature

NaHSO₄ 4 h experiments



Temparature

Na₂SO₄ 4 h experiments



Temparature





Temperature: 90°C Salt: NaHSO₄ Exposure: 4h





Temperature: 90°C Salt: NaHSO₄ Exposure: 4h



LEO 1530

WD = 12 mm

Aperture Size = 60.00 μm Signal A = QBSD Image Pixel Size = 1.172 µm

| T (0000 | | | WT% | , D | |
|--------------------------|---|------|------|--------|------|
| Temperature: 90°C | | Fe | S | Na | 0 |
| | 1 | 17.4 | 19.6 | 16.7 | 46.2 |
| Salt: NahSO ₄ | 2 | 98.7 | | | |
| | 3 | 23.3 | 20.6 | 8.6 | 47.3 |
| Exposure: 4n | 4 | 22.2 | 25.8 | 13.0 | 38.3 |
| • | 5 | 18.0 | 21.5 | 16.4 | 43.7 |
| 13 | 6 | 29.8 | 20.9 | | 48.6 |



Temperature: 90°C Salt: NaH₂SO₄ Exposure: 4h

LEO 1530

WD = 12 mm



Signal A = QBSD Image Pixel Size = 234.4 nm

| Temperature: 90°C | | | WT% | 6 | |
|-------------------|---|------|------|----|------|
| | | Fe | S | Na | 0 |
| O2HeN vite2 | 1 | 33.4 | 20.1 | | 45.1 |
| San. Natio O_4 | 2 | 95.9 | 0.3 | | 2.4 |
| Exposure: 1h | 3 | 97.0 | 0.3 | | 1.3 |
| EXPOSULE. 411 | | | | | |



Temperature: 100°C Salt: NaHSO₄ Exposure: 4h





Temperature: 100°C Salt: NaHSO₄ Exposure: 4h



Aperture Size = 60.00 µm

Signal A = QBSD Image Pixel Size = 585.9 nm



Mag = 200 X

WD = 11 mm

EHT = 15.00 kV

100µm

LEO 1530

| | | WT% | 6 | |
|---|------|------|------|------|
| | Fe | S | Na | 0 |
| 1 | 15.3 | 23.7 | 14.7 | 46.1 |
| 2 | 32.2 | 21.9 | | 45.6 |

Date :30 Sep 2014

Å



100µm LEO 1530

Mag = 200 X WD = 11 mm EHT = 15.00 kV Aperture Size = 60.00 μm Signal A = QBSD Image Pixel Size = 585.9 nm Date :30 Sep 2014





Temperature: 100°C Salt: NaHSO₄ Exposure: 4h ^{Top view}

| | | WT% | 6 | |
|---|------|-----|-----|------|
| | Fe | S | Na | 0 |
| 1 | 97.0 | | | |
| 2 | 98.2 | | | |
| 3 | 18.6 | 1.2 | 2.3 | 31.9 |



00μm LEO 1530

н

→ Mag = 200 X WD = 13 mm EHT = 15.00 kV Aper Signal A = SE2 Imag

Aperture Size = 60.00 μm Image Pixel Size = 585.9 nm



À

Temperature: 110°C Salt: NaHSO₄ Exposure: 4h



Temperature: 110°C Salt: NaHSO₄ Exposure: 4h



Temperature: 110°C Salt: NaHSO₄ Exposure: 4h

Top view



Å

Temperature: 120°C Salt: NaHSO₄ Exposure: 4h



Temperature: 120°C Salt: NaHSO₄ Exposure: 4h

Top view



Temperature: 120°C Salt: NaHSO₄ Exposure: 4h



Mag = 200 X WD = 13 mm

LEO 1530

EHT = 15.00 kV Aperture Size = 60.00 μm Signal A = SE2 Image Pixel Size = 585.9 nm Mag = 200 X WD = 13 mm LEO 1530



Temperature: 90°C Salt: Na₂SO₄ Exposure: 4h



Temperature: 90°C Salt: Na₂SO₄ Exposure: 4h

Top view





LEO 1530

WD = 13 mm

Signal A = SE2

Image Pixel Size = 585.9 nm

Temperature: 90°C Salt: Na₂SO₄ Exposure: 24h



Temperature: 90°C Salt: Na₂SO₄ Exposure: 24h

| | | WT% |) | |
|---|------|------|------|------|
| | Fe | S | Na | 0 |
| 1 | 0.3 | 24.0 | 35.5 | 40.2 |
| 2 | 62.0 | 1.1 | 4.9 | 30.8 |





Temperature: 90°C Salt: Na₂SO₄ Exposure: 24h

Top view





Temperature: 100°C Salt: Na₂SO₄ Exposure: 4h



Temperature: 100°C Salt: Na₂SO₄ Exposure: 4h

| | | WT% | | |
|---|------|-----|----|------|
| | Fe | S | Na | 0 |
| 1 | 67.1 | 0.1 | | 31.6 |

Top view



Temperature: 100°C Salt: Na₂SO₄ Exposure: 24h



Temperature: 110°C Salt: Na₂SO₄ Exposure: 24h



Temperature: 110°C Salt: Na₂SO₄ Exposure: 24h



100µm LEO 1530 Mag = 200 X

WD = 12 mm

EHT = 15.00 kV

Aperture Size = 60.00 µm Date :30 Sep 2014 Signal A = QBSD Image Pixel Size = 585.9 nm



Temperature: 110°C Salt: Na₂SO₄ Exposure: 24h



Temperature: 120°C Salt: Na₂SO₄ Exposure: 4h



| Temperature: 120°C | | | WT% |) | | |
|---------------------------------------|---|------|------|------|------|---|
| Salt: Na ₂ SO ₄ | | Fe | S | Na | 0 | |
| | 1 | 3.3 | 23.1 | 34.0 | 39.1 | S |
| Exposure: 4n | 2 | 56.8 | 0.9 | 0.3 | 33.5 | E |





Mag = 500 X WD = 11 mm

EHT = 15.00 kV

Aperture Size = 60.00 µm Signal A = QBSD Image Pixel Size = 234.4 nm

Date :30 Sep 2014



Temperature: 120°C Salt: Na₂SO₄ Exposure: 4h







.

Corrosion rate



Conclusions

- Laboratory setup worked as planned
- For Na₂SO₄:
 - corrosion seen at 90°C and 100°C, but not at 110°C and 120°C
 - Iron oxide formation
- For NaHSO₄:
 - Increasing corrosion was seen at decreasing temperature
 - Clear acid corrosion
 - Iron sulfate in pits

Conclusions

 Salts seem to absorb moisture at a slightly higher temperature than the theoretical temperatures at 60vol% H₂O. The theoretical values are not from flue gas conditions (temperature gas composition)



SUOMEN SOODAKATTILAYHDISTYS RY RAPORTTISARJA

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