

Abatement of malodorous pulp mill emissions by catalytic oxidation—pilot experiments in Stora Enso Pulp Mill, Oulu, Finland

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ABSTRACT: Catalytic oxidation can be used to abate VOC and odorous emissions from pulp mills. In this study on the catalytic incineration of chip bin emissions, we followed the operation of a catalytic pilot process by measuring emissions. The catalysts were also tested in laboratory work. It was found that catalytic incineration can treat these odorous emissions, and the conversions can even be close to complete oxidation. However, some problems occurred because of black deposits, which blocked the heat exchangers of the pilot incinerator.

Application: Catalytic incineration is a feasible technology for removing volatile organic compounds and malodorous sulfur-containing emissions at low operating temperatures.

The malodorous emissions from the typical pulp mill contain mercaptanes and other organic sulfides. They are a problem since they have foul-smelling odor at very low concentrations, even if they are not extremely toxic. With the current know-how, it is not yet possible to build a completely odorless pulp mill. Nevertheless, the odor problem may be particularly annoying if the pulp mill is located near a city center.

In the Oulu pulp mill in Finland, the undiluted malodorous gases are recovered and incinerated in the malodorous gas boiler. The resulting SO_2 is absorbed from the flue gases into caustic and is reused in the pulp bleaching process. The dilute malodorous gas-stream mixtures formed in pulp production are incinerated in the kraft recovery boiler [1]. Despite these forms of emissions treatment, some odorous gases are emitted via different parts of the pulping process. One emission source is the chip bin, where wood chips are pretreated with malodorous gases and with water vapor.

Catalytic oxidation can be used to abate gaseous emissions containing volatile organic compounds (VOC). Traditionally it has been used to treat solvent emissions. Some studies have focused on the abatement of malodorous emissions from pulp mills [2–4]. Short-term, bench-scale experiments have shown that it is feasible to use catalytic oxidation for pulp mill emissions. The big question has been about the catalyst's long-term durability, or its activity, stability, and selectivity, as impacted by sulfur

compounds. Another cause of concern is the possibility of short-term, high-concentration terpene peaks in the operating pulping process. These peaks may cause deactivation of the catalyst, which would limit the usefulness of catalytic oxidation as an abatement technology.

We carried out pilot experiments to study the use of catalytic oxidation in abating dilute malodorous emissions in the pulp mill. We selected the chip bin as the source of emissions. In many pulp mills, the chip bin is not included in the system for collecting malodorous emissions. Furthermore, it is a difficult target because the concentrations of the emission gases fluctuate so much that sometimes the explosive limit for terpenic compounds is even exceeded.

Here, we will first focus on catalytic oxidation of malodorous emissions from chip bin originating from continuous digester pre-steaming. We will also look at laboratory studies on the polymerization of terpenic compounds, since polymerization is one probable reason for the blockage of the heat exchangers.

CATALYTIC OXIDATION OF MALODOROUS COMPOUNDS

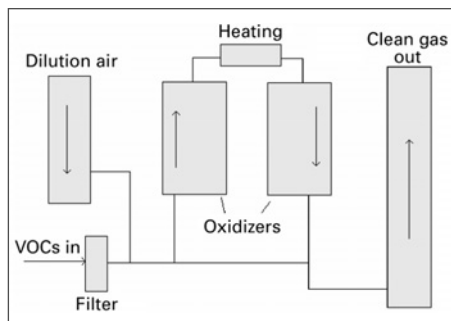
In catalytic oxidation, the organic emissions are oxidized to CO_2 and H_2O , and, in the presence of organic sulfur emissions, to SO_2 . The oxidation proceeds without flame, and the temperatures required for oxidation are significantly lower than those needed in thermal oxidation. Because the temperatures are lower, the demands for construction materials are

lower, and the treated emission concentrations can be lower in turn. In addition, the cleaning efficiencies can be over 95%, which is important for malodorous gases since they are odorous at very dilute concentrations [2, 3].

The catalytic incinerator used, presented in Fig. 1, is a regenerative “matros”-type reactor. It consists of two honeycomb systems that include catalysts and heat exchangers. The heat exchangers are considered regenerative in that they temporarily store energy from a hot gas stream. This hot gas is produced during exothermic oxidation reactions in the catalyst bed and stored in a heat sink such as that created by metal honeycombs. The hot stream is introduced into the bed until the temperature of the bed nearly reaches the temperature of the hot stream. The direction of streams is changed, and a cold gas stream is passed through the hot bed. Thus, the cold stream is heated up to the required reaction temperature by the hot bed. When the bed cools down, the directions of streams are reversed again, repeating the cycle. A regenerative heat exchanger can achieve a heat recovery effectiveness of 85% or possibly even higher [5].

This kind of operation does not need any external heating if the VOC concentrations of the emission gases are high enough. The lowest concentration needed for oxidation without extra heating, or the so-called “autothermal point,” is about 0.7 g/m^3 of VOC. If the concentration of VOC exceeds 8 g/m^3 , extra dilution air is needed to keep the emission gases under

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1. Scheme of an industrial-scale VOC incinerator.

	Conversion, %	
	350°C	420°C
H ₂ S	59.2	100
MM	72.7	100
DMS	64.5	91.9
DMDS	—*	5.1

*Higher outlet concentration.

1. Results of the catalytic oxidation of odorous sulfur compounds.

the explosion limits. In Finland, the authorities have set the limit for the maximum VOC concentration in catalytic oxidation to 8 mg/m³. The normal operating temperature of an industrial-scale catalytic VOC incinerator is about 350°C, but it varies depending on the VOC compounds to be oxidized. According to earlier bench-scale experiments, the temperature should be over 400°C in the oxidation of chip bin emissions.

In the first phase of these pilot experiments, we excluded situations that involved safety issues, such as blow-through situations on chip bins. The temperature of the chip bin was followed continuously, and if the temperature, or the rate of the temperature increase, exceeded the safety limit, then the valve in the chip bin chimney would close the inlet to the catalytic incinerator. When the chimney valve closes, the valve at the inlet of the catalytic incinerator also closes, and the system shuts down. In the incinerator, if the temperature close to the catalyst gets up to 580°C, the system begins to dilute the emission gas. When the temperature climbs to 600°C, the incinerator closes the inlet valve, and the incinerator shuts down. The catalysts used in this incinerator are metallic monoliths that will stand temperatures close to 1000°C temporarily without sintering [6].



2. Catalytic incinerator used in the experiments.

PILOT EXPERIMENTS

The pilot experiments started at the beginning of August 2003. During the pilot experiments, approximately 10% of the chip bin emissions were treated catalytically. The capacity of the used catalytic incinerator, shown in Fig. 2, was 500 m³/h. The emission stream contained mostly methanol, terpenic compounds, hydrogen sulfide (H₂S), methyl mercaptan (MM), dimethyl sulfide (DMS), and dimethyl disulfide (DMDS). The amount of terpenic compounds in the emission gas stream was roughly 40% by volume, and the amount of reduced sulfur compounds was about 60% by volume.

We observed that the emission gas at the inlet of the catalytic oxidizer included water (14 g/kg of dry gas) and that the solids content of the gas was about 0.003 kg/h. Therefore, we filtered the emission gas before introducing it into the incinerator. The composition and concentration of the emission gas stream vary with the pulping process operation. We measured the emissions of VOCs and other gaseous compounds by PID (a continuous (or in situ) Photo Ionization Detector that measures total concentrations of VOCs), by chemiluminescence, by UV-fluorescence, and by bag sampling methods. The bag samples were analyzed by gas chromatography equipped with FPD (Flame Photometric Detector, a GC detector which is used to analyze the sulphur-containing compounds).

Oxidation experiments with a Pt catalyst were first carried out at about 350°C. As Table I shows, this temperature was not high enough to oxidize odorous sulfur-containing compounds. Later, the oxidation temperature was increased to about 420°C. The conversions obtained are given in the table. The temperature was not constant, fluctuating between the upper and lower setting values.



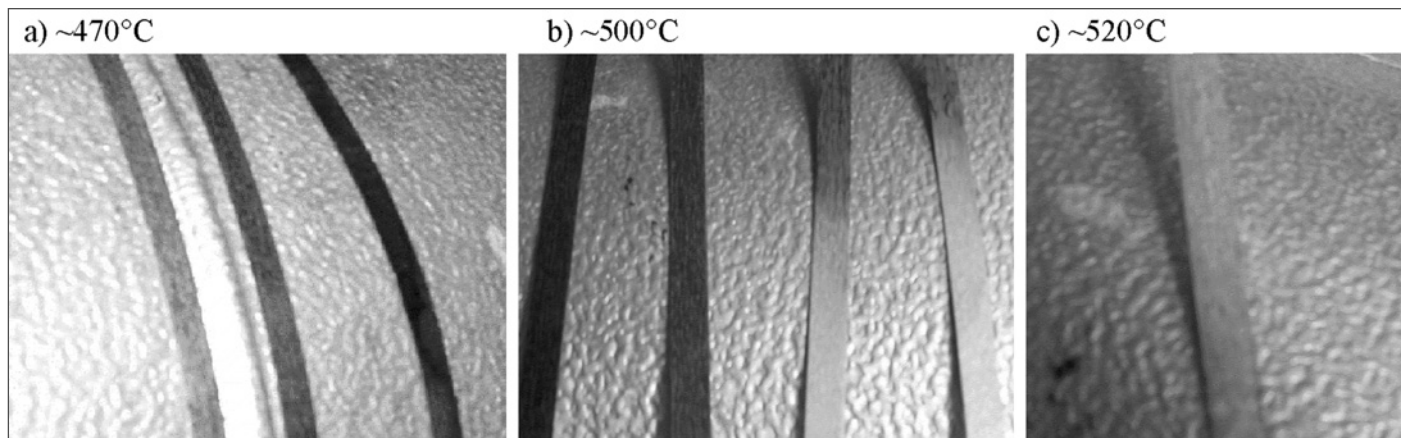
3. Black deposit that accumulated on the heat exchanger's surface.

These results indicate that the oxidation temperature should be increased further to oxidize DMS and DMDS completely. The first measurements at about 350°C showed that DMDS was formed as an intermediate during incineration. This formation was also observed in the laboratory experiments. Earlier laboratory studies carried out by Kastner *et al.*, by Martinez *et al.*, and by Dalai *et al.* showed similar results [7–10]. At temperatures close to 350°C, MM, DMS, and DMDS are not fully oxidized, and they probably form methylthiyl radicals (CH₃S) that react further to form DMDS.

Some researchers have proposed that CH₃S is one of the key intermediates of the OH-initiated oxidation of MM, DMS, and DMDS [8]. Furthermore, Kastner *et al.* have proposed a potential mechanism for the oxidation of methyl mercaptan [9]. They believe that if the R group is CH₃, oxygen is not capable of reacting with R-SS-R or R-S-R. Thus no further reaction would occur beyond this point, which accounts for the formation of dimethyldisulfide (DMDS) from methanethiol (MM) [2].

In addition, our results show that the catalytic oxidation of DMDS on a Pt catalyst needs higher temperatures than does the catalytic oxidation of DMS or MM. This finding is consistent with the results of Ok *et al.*, who carried out the oxidation of odorous compounds on a V₂O₅-WO₃/TiO₂ catalyst [11, 12]. Chu *et al.*, however, reported opposite results when DMS and DMDS were oxidized on a MnO/Fe₂O₃ catalyst [13]. This outcome may be attributable to different oxidation mechanisms on the surfaces of the different catalysts.

Later, when the oxidation temperature in pilot experiments was increased to about 420°C, the conversion of odorous compounds increased. However, the



4. Test sticks after a 1-h test run at catalytic incinerator operating temperatures of about 470°C (a), about 500°C (b), and about 520°C (c).

flow measurements showed that the flow through the system was dramatically diminished after approximately 100 h of operation. Thus, the experiments were stopped for a while. When the incinerator was opened, a black deposit was found on the surfaces. This accumulation, shown in Fig. 3, was blocking the heat exchangers.

Apparently this black deposit was being formed as a result of polymerization of terpenic compounds. Several laboratory analyses were carried out to ascertain the origin of the deposit, but the exact chemical composition of the deposit is still not well known. Thermogravimetric analysis showed first a 30% decrease in the deposit mass at about 250°C and then a 65% decrease by approximately 600°C.

In their study on the polymerization of α - and β -pinenes [14], Ramos *et al.* found that the polymerization yield of α -pinene decreases when the temperature is increased but that the polymerization yield of β -pinene is increasing at the same time. Encarnacao *et al.* [15] found that the presence of light has an important role in α -pinene polymerization, which suggests that α -pinene polymerization takes place via a radical mechanism. They also claimed that a catalyst, V_2O_5 , seems to increase the radical initiator species.

These results suggest that the temperatures used in the incinerator are not high enough to oxidize the terpenic compounds of chip bin emissions. Instead of total oxidation, the terpenic compounds are probably polymerized. It was decided that formation of deposit at different temperatures would be monitored by test sticks placed in the incinerator between the heat exchangers.

The temperature between the heat exchangers was changing according to the direction of the gas flow. In general, the lowest average temperatures were measured between the lower heat exchangers, whereas the highest temperatures were measured close to the catalyst bed. As Fig. 4 shows, the formation of the black deposit was dependent on temperature. The formation of black deposit was slightly less close to the catalyst bed at higher temperatures, compared to the formation between the lower and thus the colder heat exchangers.

This is illustrated by Fig. 4 by the fact that the sticks are placed in different places in the incinerator and the temperatures are different for each place; the most black stick has been in the coldest place and the sticks that have lighter color have been closer to the catalyst, i.e. at the warmest place. The stick presented in Frame c has been in the same place as the darkest sticks in frames a and c. The color indicates the "amount of deposit" since the sticks have been inside the incinerator an equally long time.

When the incinerator's operation temperature was increased, the formation of the deposit also decreased. However, it is possible that the heat exchangers were already blocked at the end of the experiment. In that case, the decrease in the deposit formation would result from the decreased flow-through in the system. Based on these results, more information of the deposit formation is still needed, and additional laboratory studies will be required.

LABORATORY EXPERIMENTS

In addition to the deposit formation studies, catalysts were tested in the laboratory

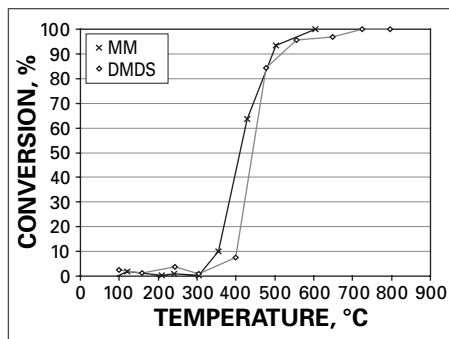
to elucidate the activity of the catalyst in the turpentine oxidation. Because emissions from the chip bin contained different terpenic compounds (mostly α - and β -pinenes), we chose French turpentine as a model compound. French turpentine is made from pinewood, and it contains mostly α - and β -pinenes but also some minor constituents such as limonene and camphene.

In the experiments, we monitored only the conversions of α - and β -pinenes. In addition to the activity experiments (light-off tests), we also tested the catalyst with different space velocities and with different turpentine concentrations close to T_{100} , where T_{100} is defined as the temperature of 100% conversion. Oxidations of MM and DMDS were tested without a catalyst.

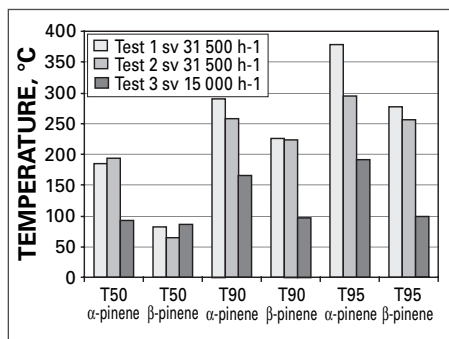
In the activity tests, the GC/FID was calibrated with α - and β -pinene. (FID, or flame ionization detector, is a GC detector which can be used to measure organic compounds. In this case, sulphur containing compounds are analyzed by FPD and other organic compounds by FID.) Since the used model compound was French turpentine, the calibration was carried out with that specific turpentine and not with pure compounds. However, the other data needed in the calibration were for the pure compounds. This dilemma may have led to some systematic error. However, the systematic error is the same in all the experiments, and the results can therefore be compared with each other.

The catalyst used in the laboratory experiments was a monolithic Pt catalyst. This catalyst was designed especially for sulfur-containing conditions, and the same catalyst was used in the pilot-scale

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5. Light-off curves for MM and DMDS without any catalyst.

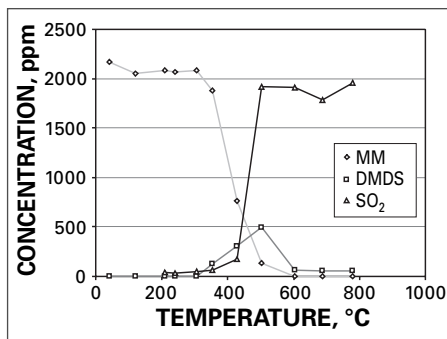


8. T50, T90, and T95 temperatures for α - and β -pinene with different space velocities. Tests 1 and 2 show the repeatability of the experiments.

experiments. The laboratory experiments were carried out in a quartz reactor. The reactor was heated in a tube oven from room temperature to about 800°C. The temperature was measured before and after the catalyst (at the catalyst inlet and outlet).

The piping between the feeding system and the reactor and from the reactor to gas chromatography was made of Teflon® to minimize the possible adsorption of the VOC compounds on the lines. The turpentine was fed with a syringe pump and a vaporizer, where the turpentine vapor was mixed with air from a mass flow controller.

The outlet gas flow was analyzed by the GC/FID. The MM, DMDS, and turpentine concentrations in the experiments were 2000 ppm. The concentration of α -pinene in turpentine was around 1650 ppm, and the concentration of β -pinene was approximately 350 ppm. In general, a space velocity of about 31,500 h⁻¹ was used in the experiments, which was the same as the theoretical space velocity in the pilot-scale incinerator.



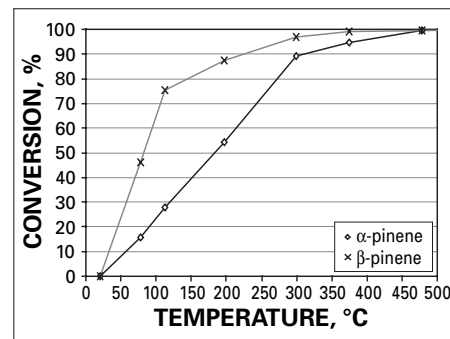
6. Concentrations of MM, DMDS, and SO₂ during the noncatalytic oxidation of methyl mercaptan.

RESULTS OF LABORATORY EXPERIMENTS

In the activity measurements, each conversion was determined as a function of temperature (light-off curves). The conversions of the noncatalytic oxidation of MM and DMDS are presented in Fig. 5.

As Fig. 5 shows, the noncatalytic conversions of MM and DMDS does not exceed 20% at 350°C. At 420°C MM conversion is close to 60% whereas for DMDS it is close to 30%. Corresponding conversions in pilot-scale experiments (catalytic) were significantly higher (Table I). Reportedly, it requires operating temperatures of 760–870°C for regenerative thermal oxidation to abate odorous emissions [7]. The results presented here are consistent with that observation and with the results of the blank tests with DMDS made by Chu *et al.* [13]. Catalytic oxidation decreases the temperatures required to oxidize MM and DMDS. A catalyst is therefore critical at oxidation temperatures of 350°C and 420°C. The formation of intermediates during the oxidation of organic compounds may occur in both thermal and in catalytic incineration [16]. As mentioned before, the possible formation of DMDS was observed during catalytic incineration of MM when the operation temperature was 350°C. Figure 6 shows the decrease of MM concentration and the formation of DMDS during the noncatalytic oxidation of MM.

In addition to DMDS, small amounts of other intermediates were detected during the oxidation of MM. These results suggest that the formation of DMDS from MM decreases when the oxidation temperature is increased. Furthermore, the formation of DMDS from MM is not an entirely catalytic phenomenon. In general, the addition of the



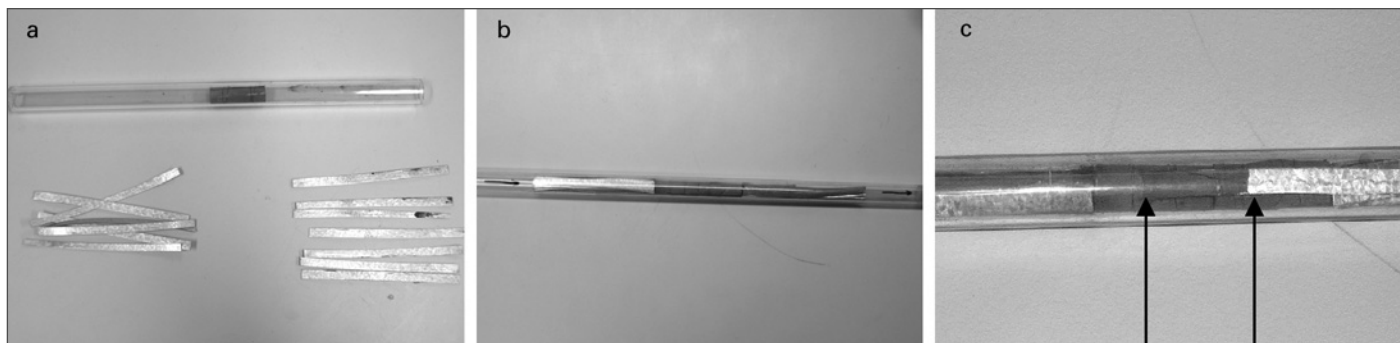
7. Light-off curves for α - and β -pinene oxidation on a Pt-catalyst with the space velocity of 31,500 h⁻¹.

catalyst decreases the T₅₀ and T₁₀₀ temperatures, which means that the temperature required for the total oxidation of MM on a catalyst is less than approximately 600°C.

Our preliminary experiments showed that the deposit was formed close to the catalyst outlet, not at the inlet. As a result, the polymer probably forms because of incomplete oxidation of terpenic compounds. Therefore we studied the activity of the catalyst in oxidation of terpenic compounds.

The light-off curves for α - and β -pinene catalytic oxidation are presented in Fig. 7. The light-off temperature (T₅₀) for α -pinene is about 190°C and is about 80°C for β -pinene. In general, the T₅₀ of β -pinene is lower than that of α -pinene, which we also observed in the activity tests with other catalysts. In these experiments, only the concentrations of α - and β -pinene were monitored and quantified. Mass balances were not calculated, so it may be possible that the decrease in the concentration of α - and β -pinenes may also be attributable to reactions other than oxidation (such as polymerization).

In addition, we studied the effect of concentration and space velocity on the catalytic oxidation of α - and β -pinene. In general, a decrease in the space velocity decreases the T_x temperatures of both α - and β -pinene oxidation, as Fig. 8 shows. This response would suggest that a more complete oxidation of those pinenes is possible with the smaller space velocities. Smaller space velocities may decrease the formation of polymer deposits in the industrial-scale applications. However, this hypothesis has not been entirely proved, since the polymer cannot be analyzed with the existing laboratory facilities and the mass balances have not been calculated. The mass bal-



9. Deposit formation in (a) 4 h with one catalyst, (b) 13 h with two catalysts, and (c) 13 h with two catalysts, where one test stick shows the formation of the deposit decreases after about 1.5 catalyst.

ances could not be calculated because the complicated turpentine mixture prevented us from quantifying all of the intermediates.

The deposit formation was studied in the laboratory experiments. A short experiment with construction material plates and a catalyst showed the formation of a black deposit on the plates in 4 h with the space velocity value of 31,500 h^{-1} . The formation of the black deposit was not observed in a 4-h test with the space velocity of 15,000 h^{-1} . However, this experiment is not very accurate since it is only possible to observe the deposit formation visually. The deposit was formed on the plates after the catalyst, but not before. The deposit changed its color from black to light brown after the plates were taken off the reactor and left in the open air on a table. **Figure 9** shows the results of these tests.

These results support the conclusion that the black deposit formation is caused by the incomplete oxidation of terpenic compounds. **Figure 9c** shows that the black deposit forms close to the front edge of the catalyst bed. However, it is not found at the end of the catalyst bed. In the pilot-scale incinerator, the black deposit formation was more significant in the cooler heat exchangers. This means that the gas stream at the outlet of the catalyst probably includes reactive terpenic species that start to polymerize when the gas stream cools down at the lowest heat exchangers, blocking the coolest heat exchangers more predominantly.

Figure 10 presents the effect of turpentine concentration on the oxidation of pinenes close to T_{100} (about 300°C). In these experiments, we used a constant space velocity of about 15,000 h^{-1} . The increase in the concentration of pinenes does not significantly affect the end conversions, as the figure shows.

Based on these experiments, the space velocity appears to have a more significant effect on these oxidation reactions than on the concentration of pinenes. This finding was also observed in the preliminary solvent-VOC oxidation experiments [16].

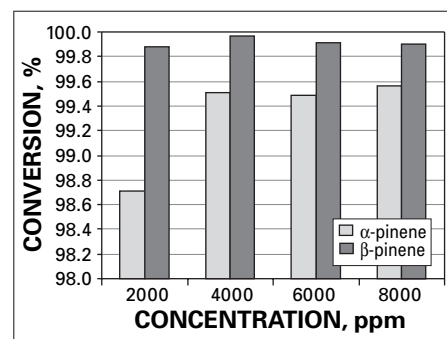
When the conversions achieved in the laboratory scale are compared to those of the full-scale operation, it is important to remember that the two are not quite the same. With a dual-bed regenerative catalytic incinerator, it is not possible to achieve conversions of 100% because of valve leakages and other losses that are quite difficult to control. However, it is possible to avoid losses of this kind by employing a triple-bed construction.

CONCLUSIONS

The abatement of chip bin emissions is a complicated and demanding task. The emission concentrations vary from very dilute to very concentrated mixtures. In addition, they include volatile organic compounds, odorous sulfur compounds, organic terpenic compounds, solids, and water vapor.

It seems that catalytic oxidation is not by itself a proper method to treat these emissions. Additional filtering of solid particulates and the possible drying of the emission gas stream are needed. At this stage, the problem does not appear to be the deactivation of the catalyst by sulfur-containing compounds but the deposit formation on the heat exchanger's surfaces, which may be caused by the presence of terpenic compounds. To solve this problem, we carried out some laboratory experiments.

In the emissions of the Oulu pulp mill, the concentration of pinenes varies considerably. The maximum concentrations may be much higher than those



10. Effect of turpentine concentration on the end conversion in α - and β -pinene oxidation.

used in the laboratory experiments. A concentration of 8000 ppm of pinenes is about 49 g/m^3 . However, the safety limit for the operation of a catalytic incinerator is 8 g/m^3 , and the emission concentrations will be diluted above this limit value. Based on this consideration, it may be concluded that a decrease in the inlet concentration does not diminish the possibility that polymer may be forming in this case.

The decrease in the space velocity decreases the T_x temperatures. At the same time, the oxidation is more complete since emission compounds have enough time to react on the catalyst. From these experiments, we cannot exclude the possibility that polymer deposits form. The visual observations indicate, however, that the formation of a polymer deposit is decreased when lower space velocities are used. The experiments in the pilot plant showed that the formation of a polymer deposit was faster at lower temperatures. This condition indicates that the operation temperature should be increased.

The pilot experiments will continue with the decreased space velocity within the catalytic incinerator. The operation temperature will be increased, and the

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drying of the emission stream will be carried out. The drying device will also filter the concentration peaks that may cause overheating of the catalytic incinerator. Drying will also decrease the possible cooling effect in the lower heat exchangers as a result of water vaporization. **TJ**

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INSIGHTS FROM THE AUTHORS

In general, odor abatement is a motivating topic of research. In Oulu in particular, it is a very interesting research problem, since the pulp mill is located in close proximity to the city center. Partly due to public opinion and the image of the whole city, partly due to environmental interest, the pulp mill is strongly supporting this kind of research.

Previously, catalytic oxidation has been used in solvent emissions abatement; it is a topic we have studied for some years. The abatement of odorous gases is a possible extension of the application for the existing technology.

Prior to experiments, it was assumed that the catalyst ageing or poisoning would be the largest difficulty. Presently, the biggest challenge seems to be the fouling of the heat exchangers that must be overcome before studying the catalysts durability. This research has been, and still is, very challenging. After one solves one

problem, the next surprise will appear. This research has brought up a great number of new ideas concerning further studies and applications.

This paper is of benefit because it gives new information about the catalytic incineration, experiences from the pilot experiments in odor abatement, and discusses the benefits and pitfalls of the technology. The next step in research is to overcome the heat exchanger's fouling problem and to continue the research about the catalyst's durability.

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