

Investigation performed to predict the SO_2 and HCl emissions of rotary kilns have shown that the chemical reactions needed to assess these emissions also enable quantification of the generation rate of build-ups in the preheating zone of these kilns. As anticipated by the theory and confirmed by industrial tests, sulphur and chlorine loops were found. The chlorine loop is closely interconnected to the sulphur loop that is itself influenced by the process conditions prevailing in the kilns. The quantification of the thus resulting emissions and generation of build-ups requires a precise assessment of all reactions involving sulphur and chlorine. However the first results of the modelling showed only a fair agreement between calculations and monitoring results. Further investigations were therefore carried out. Accordingly it was found that potassium plays a key role in the chemical reactions occurring in rotary lime kilns. The inclusion of this element in the modelling enabled a significant increase in the accuracy of the prediction. This paper presents the chemical reactions used in the model.

TEXT Dr.-Ing. Thomas Schlegel, Dipl.-Ing. (ENSCCF) Guilhem Padox, EESAC SAS Duingt/France

EESAC SAS

Modelled fate of sulphur and chlorine in rotary lime kilns

Part I: Chemical equilibria prevailing in the kilns

1 Introduction

The lime industry is using rotary kilns especially for the flexibility of this production tool. The energy efficiency of rotary kilns was along the time improved, especially with the development of shaft or vertical preheaters.

Practical experience shows that operators of rotary lime kilns face several challenges:

- » Rotary kilns equipped with vertical preheaters have to be frequently stopped since the preheaters are clogging for reasons that were

until now unexplored and often considered as unavoidable. These stops trigger operational costs (cooling down of the kiln, cleaning of the preheater, heating up of the kiln, etc.) and production and operational losses

- » Full compliance with the air emission limits (in particular SO_2 and HCl) has to be ensured
- » The quicklime has to comply with customer quality requirements (e.g. sulphur content in the lime)

These challenges are in fact interlinked and require a detailed understanding and description of the chemical reactions occurring at various places in the kilns.

In this context, EESAC has performed extensive research work. Its main objective was to develop a predictive tool (CheMoCal v.2 software) for the lime industry enabling:

- » To identify the process conditions that increase or reduce the potential clogging of shaft pre-heaters and consequently reduce the costs linked to production stops
- » To predict the HCl and SO₂ emission levels of rotary kilns using different types of fuels and operated under various conditions

This article describes the theoretical chemical background that was used in the model whereas the next publication (to be issued) will present some key results from the modelling.

2 Behaviour of sulphur and chlorine compounds in rotary lime kilns

2.1 Generation of SO₂ emissions

High sulphur contents can represent a major quality issue in the lime industry in particular when lime is used in the steel industry. For this reason, sulphur is an element that is continuously traced and analysed. The sources of sulphur in a lime kiln are:

- » The impurities of the stone used to feed the lime kiln
- » The fuels used for the calcination of lime, e.g. coal, petcoke, alternative fuels, etc.

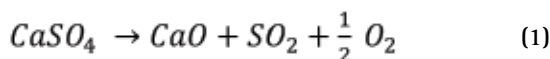
In the raw material (limestone) sulphur is usually found in the form of calcium sulphate (CaSO₄). However in some deposits sulphur can be associated with barium (in the form of barium sulphate BaSO₄) or can be present in a reduced form, e.g. as iron or calcium sulphide (respectively FeS and CaS). In fossil fuels like coal or petcoke [1, 2, 3, 4] sulphur is found in the form of metallic sulphides (FeS, ZnS, PbS), organic sulphides, thiophene, sulphates (CaSO₄, BaSO₄) or as elemental sulphur.

Depending on the speciation of sulphur forms that are introduced in the lime kiln, and also on the chemical and physical conditions (temperature) prevailing in the different areas of the kiln, sulphur can be potentially chemically converted and generate SO₂ emissions.

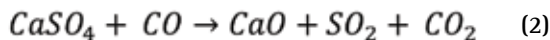
The main chemical reactions and conditions leading to SO₂ emissions in rotary lime kilns are described in the following sections (from 2.1.1 to 2.1.3).

2.1.1 Calcium sulphate

Under oxidising conditions and high temperatures, calcium sulphate can be decomposed through the following reaction:



Under reducing conditions, calcium sulphate is decomposed and leads to the formation of SO₂, with reaction temperatures depending on the partial pressure of CO and SO₂.



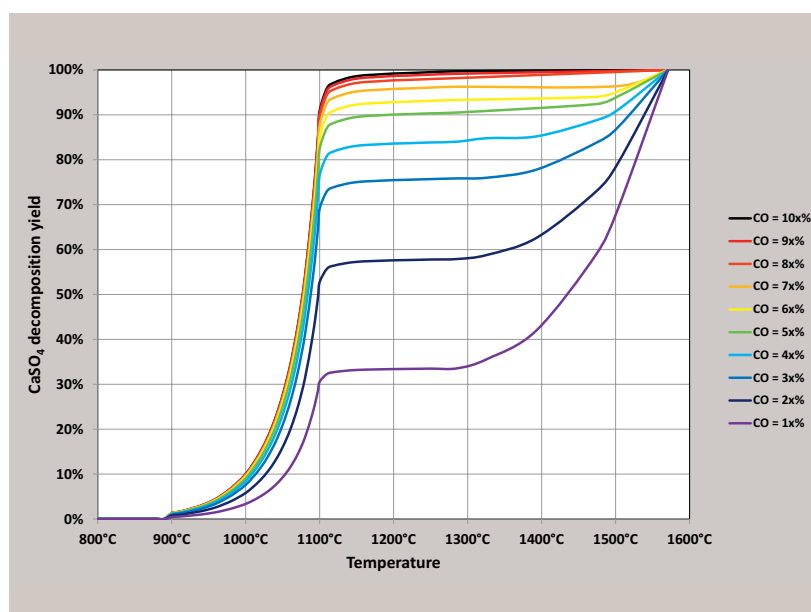
Equilibrium calculations performed with FactSage then enabled precise calculation of the calcium sulphate decomposition temperature under various conditions. These calculations showed in particular that:

- » Rising CO concentrations drastically reduce the calcium sulphate decomposition temperature from approximately 1300°C (in the absence of CO) to levels ranging between 600°C to 750° (at a CO level of 0.5%)
- » Higher CO concentrations decrease even more the decomposition temperature of calcium sulphate
- » High initial SO₂ concentrations in the kiln gas (due for example to the combustion of sulphur rich fuels) increase the calcium sulphate decomposition and thus inhibit the decomposition of calcium sulphate

Furthermore the above calculations provided quantitative information about the decomposition yield of CaSO₄. Thus [Figure 1](#) displays the relationship between this yield, the temperatures and the CO levels.

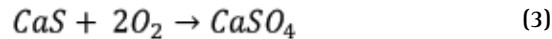
From [Figure 1](#) it becomes clear that the qualitative assessment of the CaSO₄ decomposition in the lime kiln is more complex than next expected, thus requiring an accurate thermal and chemical modelling.

1 Decomposition yield of CaSO₄ at different temperatures and different CO levels



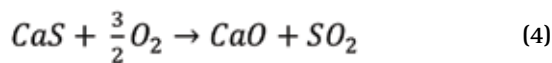
2.1.2 Sulphides

Sulphides introduced in a rotary lime kiln are generally fully oxidised. Calcium sulphide reacts at temperatures exceeding 500°C according to the following reaction:

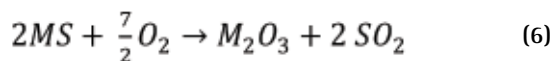
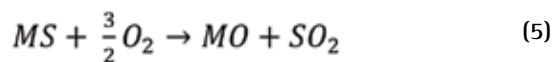


The calcium sulphate produced in this reaction can be decomposed in the hotter areas of the kiln and thus finally generate SO_2 as described by (1) and (2).

At temperatures exceeding 1320°C, the oxidation of calcium sulphide can directly produce SO_2 as shown below:



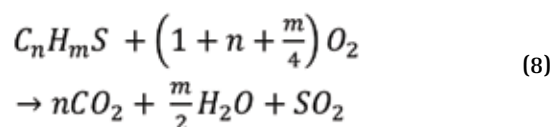
In a similar way, metallic sulphides are oxidised above 500°C and lead to the release of SO_2 as described in the following reactions.



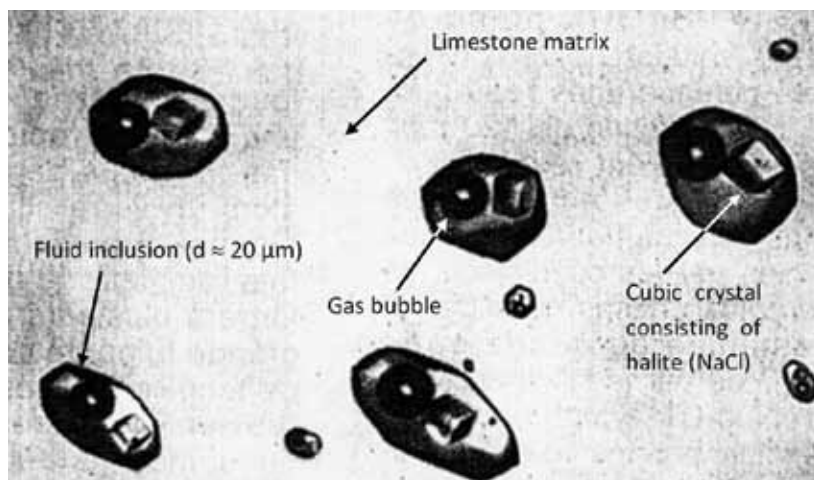
From the above equations, it can be concluded that sulphides introduced in rotary lime kilns always generate SO_2 .

2.1.3 Elemental and organic sulphur

Both of these sulphur species lead to the formation of SO_2 in the kiln as described in (7):



2 Fluid inclusions inside a limestone matrix detected with a scanning electron microscope (SEM)



2.1.4 Summary of SO_2 generation in a rotary lime kiln

The sulphur fed into the kiln through the fuels is almost fully converted into SO_2 in the vicinity of the burner. The other sulphur species fed through the stone feed may be converted into SO_2 . The yield of the conversion depends on the process conditions (oxidising vs. reducing), the original SO_2 concentration in the kiln atmosphere and the temperature levels in the burning zone. The thus generated SO_2 can be fully or partially emitted depending on the conditions prevailing in the preheating area.

2.2 Generation of HCl emissions

In lime kilns, most of the chlorine input is usually due to the mass of chlorine fed through the stone feed. The chloride content of limestone ranges from few parts per million (ppm) up to several hundreds of ppm. Investigations made about the origin of the chloride show that this element is directly linked to small fluid inclusions present in the limestone matrix as displayed in Figure 2.

Since the size of the inclusions is in the order of magnitude of several micrometers, it is not possible to sample their content for a chemical analysis. However thanks to recent physical measurement techniques [5, 6], the composition of these fluid inclusions can be indirectly determined. For limestone, following chloride species are found (sorted by decreasing importance of concentration):

- » NaCl or halite that can be easily identified in the inclusions because of its cubic crystals. From the literature it can be estimated that the chloride in form of NaCl represents 80% to 85% of the chloride content measured in the limestone
- » $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$ or bischofite, the chloride associated to this species represents approximately 10% to 15% of the total chloride content
- » Traces of KCl (sylvite) and CaCl_2

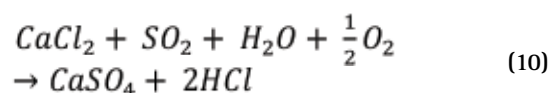
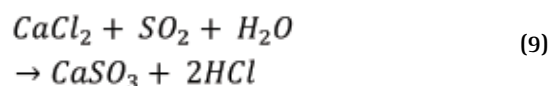
Following an extended literature review about the chlorine species found in fuels, it can be concluded that most of the chlorine is associated to NaCl, MgCl_2 and organic species in particular when waste fuels are burned.

2.2.1 Behaviour of NaCl and KCl

In the thermal and chemical conditions prevailing in the flue gas of lime kilns, all thermodynamical calculations carried out with FactSage demonstrated that NaCl and KCl are never decomposed by other substances (O_2 , H_2O , SO_2 etc.) so as to generate HCl emissions. These stable compounds are partly liquefied or vaporised in the hotter zones of the kiln and leave the kiln either through the quicklime or through the flue gas. In the cooler areas of the kiln (e.g. in the preheating zone), they may condensate and be brought back to the kiln together with the limestone.

2.2.2 Behaviour of CaCl₂

In the sole presence of water (vapour), thermodynamical calculations show that calcium chloride is thermally stable up to a temperature of about 800°C. However in the presence of SO₂, water and oxygen, it can react at temperatures above 290°C as described hereafter:

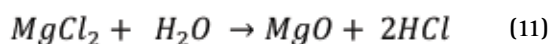


The yield of the thermal decomposition of calcium chloride according to (10) is presented as an example shown in Figure 3.

Figure 3 emphasizes that, in the presence of small amounts of SO₂, calcium chloride can be already totally decomposed below 500°C and thus generate HCl emissions. Above this temperature, the decomposition yield starts to decrease but remain significant up to 1400°C.

2.2.3 Behaviour of MgCl₂

Magnesium chloride is the less stable chloride species found in the fluid inclusions. First of all, it can be thermally decomposed at temperatures above 200°C in the sole presence of water as shown below:

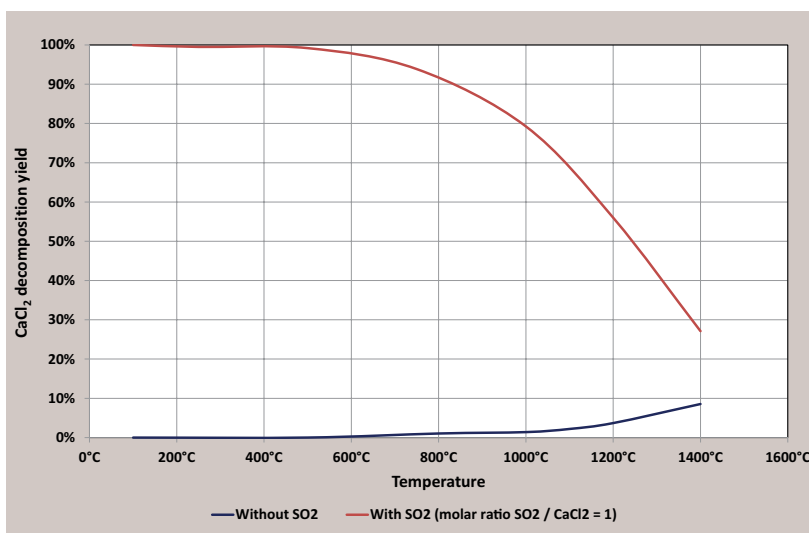


Since the magnesium chloride is always associated with water in the fluid inclusion, the above reaction indicates that as soon as the limestone temperature exceeds 240°C in the preheating zone of the kiln, HCl emissions can occur.

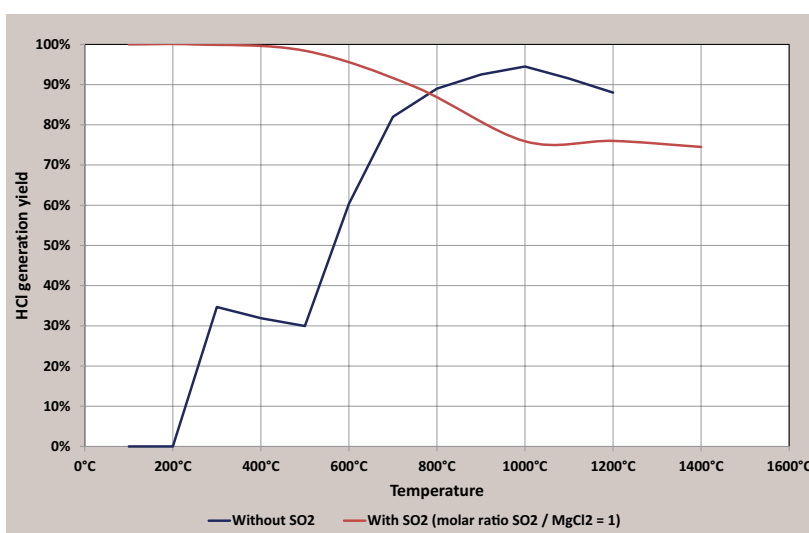
Furthermore magnesium chloride can react in the same way as calcium chloride is doing in the presence of SO₂, water and oxygen at temperatures exceeding 180°C.

The yield of HCl that is generated during the thermal decomposition of MgCl₂ is displayed in Figure 4.

As shown in the above figure, magnesium chloride is almost totally decomposed in the presence of SO₂ within the temperature range (from 100–1400°C). Without any SO₂, the HCl generation yield increases in a first step with the temperature, before remaining almost constant between 250 and 350°C. This stabilisation is explained by the formation of a stable compound (MgClOH) in this temperature window. Above 350°C this intermediate compound decomposes and thus generates HCl emissions.

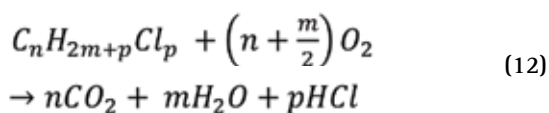


3 Decomposition yield of CaCl₂ at different temperatures and SO₂ levels



2.2.4 Organic chlorine

Chlorinated organic compounds are fully destroyed in the kiln according to the following generic reaction that also releases HCl.

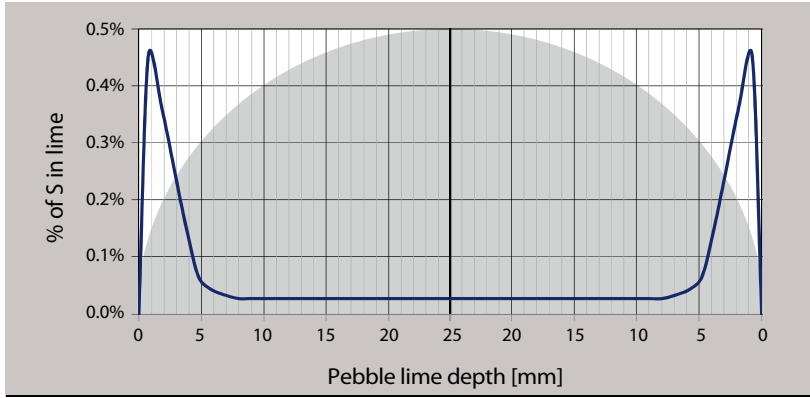


2.2.5 Summary of HCl generation in a rotary lime kiln

In the conditions prevailing in lime kilns sodium chloride (NaCl) that represents the main chlorine input through the stone feed remains fully stable even at high SO₂ levels compounds. The behaviour of KCl is the same. Therefore it can be considered for the modelling that none of these contribute to the generation of HCl. Conversely the organic chlorine fed into the lime kilns through the fuels is fully converted into HCl.

As demonstrated by the thermo-chemical calculations magnesium chloride (that represents the

4 HCl formation rate from the thermal decomposition of MgCl₂



5 Sulphur concentrations in the cross section of a 50 mm lime pebble

second most important chlorine input through the limestone) and calcium chlorides have the ability to generate HCl emissions especially in the presence of SO₂. Because of this property, the chlorine fate in lime kilns cannot be considered separately from the sulphur one. Both are connected and require a holistic approach for the modelling.

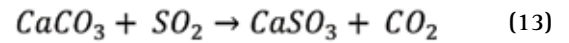
As the generation of SO₂ is promoted by reducing conditions (according to (2)), this SO₂ favours the decomposition of magnesium and calcium chloride. As a result, it can be anticipated that reducing conditions in lime kilns promote the generation of HCl emissions if all remaining process parameters and inputs remain constant. This assumption is in fact validated by numerous measurements carried out on lime kilns as discussed in the following.

2.3 Capture of SO₂ and HCl emissions in a rotary lime kiln

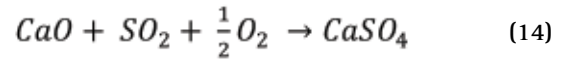
2.3.1 SO₂ capture

In the preheating zone of the lime kiln, SO₂ generated in the kiln is able to react with calcium carbonate and calcium oxide according to the following reactions:

» At temperatures between 480 and 925° C:



» At temperatures above 900° C:



The yield of these reactions depends especially on:

- » The potential for SO₂ to be in close contact with CaCO₃ or CaO (either in the form of pebbles or fine particles)
- » The temperatures in particular in the preheating zone of the kiln where the capture can occur
- » The conditions in the kiln atmosphere, as reducing conditions favour the decomposition of calcium sulphate, and thus hinder the capture of SO₂

In order to assess the amount of SO₂ that is captured by the limestone/lime pebbles in the preheating zone, special measurements were performed: limestone pebbles were heated up to 1000° C and exposed to a gas stream whose major components were typical for the flue gas composition of lime kilns. The SO₂ concentration of this gas was 200 mg/Nm³. In the following step the sulphur concentrations at different depths of the pebbles were determined.

It was thus found that sulphur is concentrated in a nearly 5 mm thick external layer of the quicklime. The thickness of this layer is independent of the size of the pebbles as it represents the diffusion depth of SO₂ into the lime mass. At greater depths, the sulphur concentrations decrease and reach finally a level that corresponds exactly to the amount of sulphur that is initially present in the limestone. An example of the monitored distribution of the sulphur concentrations in a 50 mm pebble (considered in the picture as an ideal sphere) is shown in Figure 5.

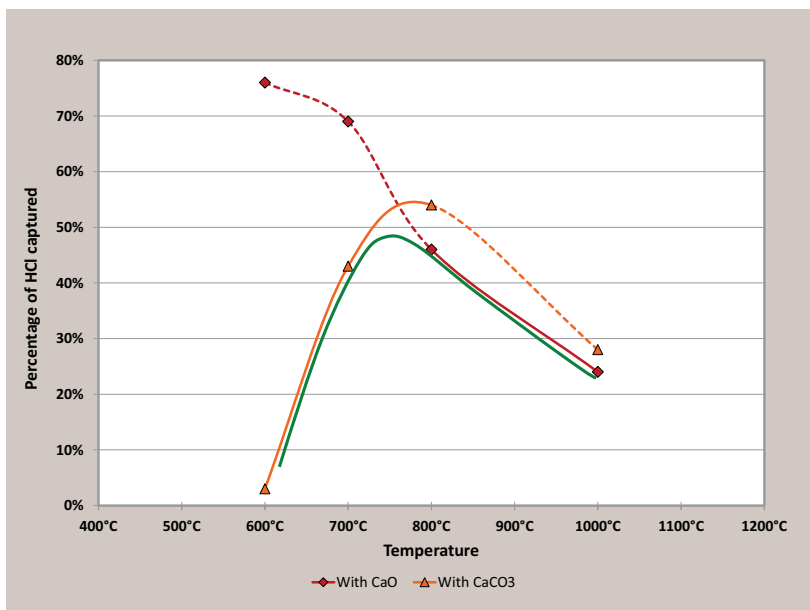
Although the size of the diffusion depth is relatively small compared to the size of the pebbles, the volume of this external layer is in proportion much more important as it represents 25 % of the pebble volume for 50 mm pebbles and 43 % for 30 mm pebbles.

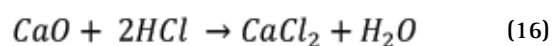
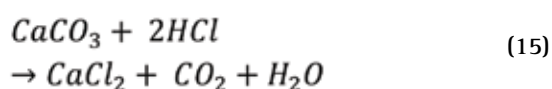
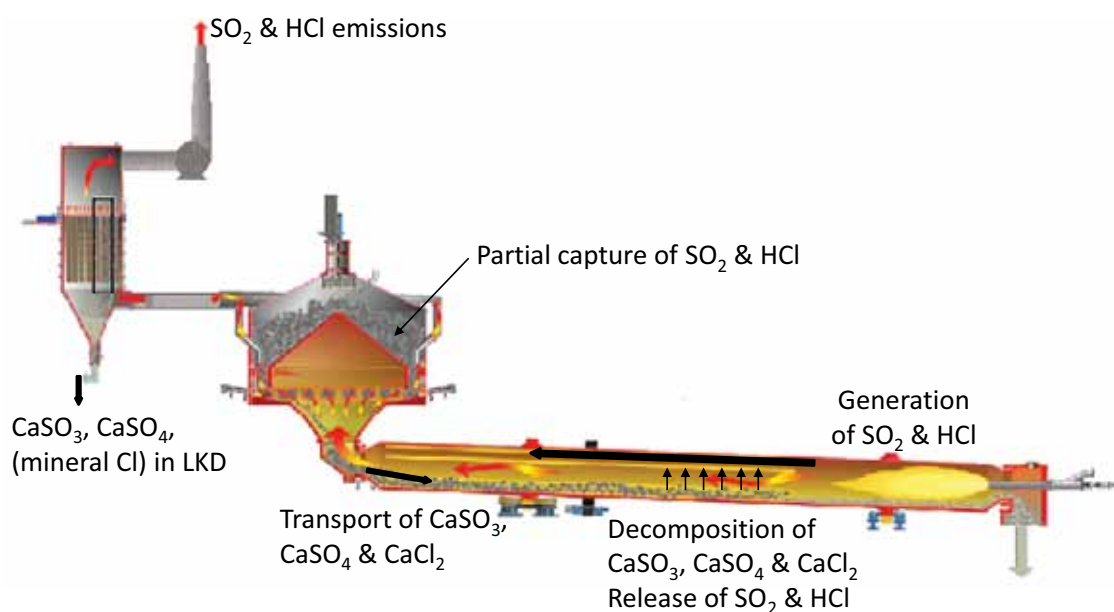
This experiment demonstrated finally the ability of lime pebbles to react chemically with the kiln atmosphere is mainly driven by the available surface exchange between the lime particle/pebble and the kiln gas. Consequently small pebbles or fine particles have then a better ability to chemically react with gases present in the kiln.

2.3.2 HCl capture

As for SO₂, HCl is able to react with the lime and the limestone present in the preheating zone of the kiln as shown below:

6 HCl capture by CaO or CaCO₃ at different temperatures according to [7]





Measurements aiming at measuring the capture of HCl by limestone or lime pebbles could not be carried out as was previously shown for SO₂. Based on the respective physical properties of HCl and SO₂, it was assumed in the following that the diffusion of both gases into the pebbles being quite similar. Therefore the same diffusion thickness was assumed in the model for HCl and SO₂. The main difference is that the yields of the above reactions (15) and (16) are different from those of SO₂.

Based on the laboratory study carried out by [7]¹ with CaCO₃ and CaO, the temperature-depending yields of the above reactions could be assessed. The experimental results are summarized in orange and red in Figure 6.

From Figure 6 it follows that the yield of the HCl capture by calcium species is the highest in the kiln preheater area (i.e. at temperatures below 900°C). For the modelling the green curve was used as a proxy for calculating the HCl capture ratio as a function of the temperature.

2.4 Zones of reactions

The chemical reactions described in sections 2.1 to 2.3 will occur at different areas of the kilns as briefly described hereafter:

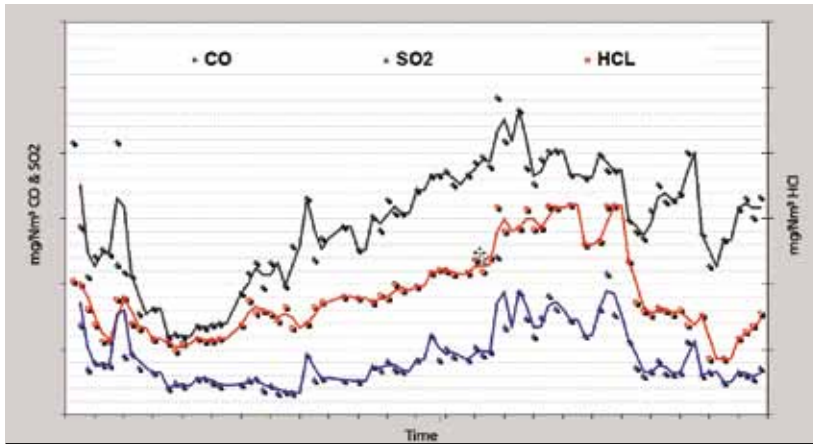
- » In the inner part of the kiln, SO₂ is generated in the vicinity of the burner during the combustion of the fuels or when the calcium sulphate fed

through the stone feed is decomposed (especially under reducing conditions)

- » This SO₂ leaves the kiln with the flue gas and enters into contact with the pebbles in the preheating zone. Part of it is captured on the pebbles; another part is sorbed on the fine particles that escape from the preheater together with the remaining gaseous SO₂
- » The sulphur loaded particles are then removed from the flue gas in the filter whereas the (gaseous) SO₂ is discharged into the atmosphere
- » The pebbles that were enriched with sulphur in the preheating zone move to the kiln. There, the calcium sulphate / sulphite accumulated in the external layer of the pebbles can be decomposed, thus releasing SO₂

As a consequence, a sulphur loop is created as shown in Figure 7. Such loops are also found in travel grate rotary kilns and to a less extent in straight rotary kilns. The fate of HCl in these kilns is to some extent comparable with the one described for SO₂ accordingly a chlorine loop is created too as shown in the following figure. Both loops are connected as the SO₂ levels in various areas of the kiln influence the release or the capture of HCl.

From the above presented chemical reactions it can be already seen that process conditions (in particular reducing conditions) influence the SO₂ levels in the kilns. These, in turn, impact the HCl levels. Therefore it can be expected that for constant sulphur and chlorine inputs, the SO₂ and the HCl emissions might be, to some extent, correlated with the CO levels. This link is indeed occasionally observed as shown in Figure 8.



8 Continuous emission monitoring of the CO, SO₂ and HCl emissions of a lime rotary kiln

However, the above described correlation is in many cases not so clear, especially between HCl and SO₂ or CO. In addition preliminary model calculations taking into account the above described reactions have shown that the agreement between calculated and monitored emissions and generation of build-ups was only fair. For these reasons, further research was needed so to identify additional element(s) and reaction(s) that increase the accuracy of the prediction.

3 Role of potassium in lime kilns

The technical literature dealing with the cement process (e.g. [8, 9]) describes in detail the alkali loops existing in cement rotary kilns. The alkalis participating in these loops are identified mostly in the form of chlorides (sodium and potassium chlorides). According to the literature the introduction of potassium (especially in the form of potassium sulphate) inside cement kilns leads to a significant decrease of the chlorine content of the clinker, as alkalis (especially potassium chloride) are released via the kiln atmosphere in the area of the kiln burner [10].

In the lime industry, the role of potassium in the process was not investigated very much until

now. To the knowledge of the authors, there is only one publication explaining very briefly the interactions between potassium and chlorine in a rotary kiln [11]. It was therefore worth investigating the origin and the role of potassium on the sulphur and chlorine loops in the lime process.

3.1 Sources of potassium in lime rotary kilns

3.1.1 Stone feed

As mentioned in section 2.2, fluid inclusions that are found in the stone matrix may include traces of sylvite (KCl). A potassium input through the limestone is therefore possible.

Furthermore in some quarries, due to the local geological conditions, the blasting and mining operations lead to a mixing of clay with limestone. This clay consists mainly of potassium feldspar whose generic chemical composition is $K_x Na_y Ca_{1-(x+y)} Al_{2-(x+y)} Si_{2+(x+y)} O_8$. This clay is sometimes removed in trommel screens or in washing installations. However if the clay is not removed, it enters into the kiln simultaneously with the limestone, thus increasing drastically the potassium input through the stone feed.

3.1.2 Fuels

In fossil fuels, the potassium species are generally potassium chloride (KCl), potassium sulphate (K₂SO₄) or potassium sulphide (K₂S) [12].

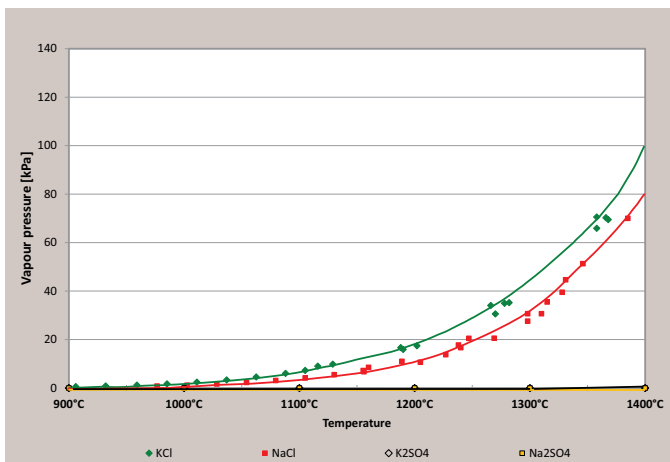
3.2 Behaviour of potassium species

3.2.1 Behaviour of KCl

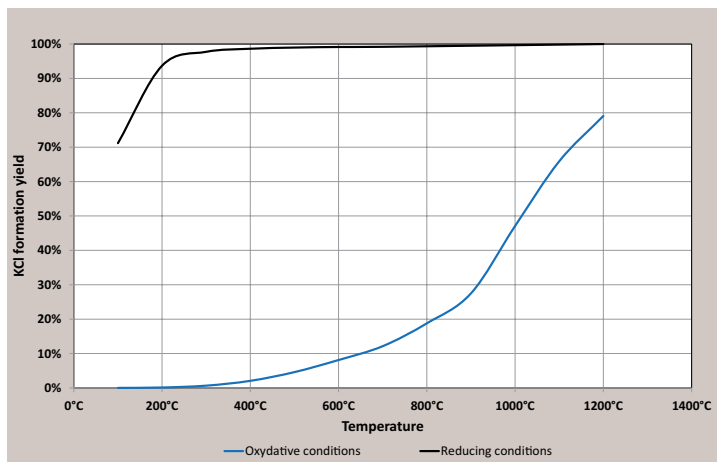
As already described in section 2.2.1, KCl is a very stable compound that decomposes neither thermally nor chemically in the conditions prevailing in lime kilns. The compilation of various physical databases leads to the conclusion that potassium chloride has the highest volatility among the different chlorine or sulphate salts found in lime kilns.

As a result of its high volatility potassium chloride, once formed in the hot areas of the kiln, can

9 Vapour pressures of different salts found in lime kilns as a function of the temperature



10 KCl formation yield from K₂SO₄

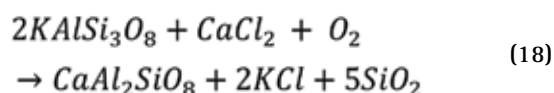
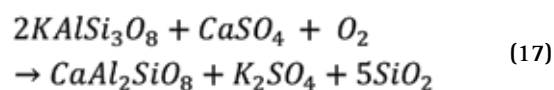


leave the kiln as vapour. In the preheating zone the flue gas is quickly cooled to temperatures below 770°C (i.e. the melting point of KCl). Hence potassium chloride can condensate or eventually crystallise either in the cool areas of the preheater or on the limestone pebbles. In the first case, the liquid KCl will act as glue for the dust particulates flowing in the flue gas, thus generating build-ups in the preheater. In the second case, the liquid KCl will move back to the kiln with the pebbles thus creating a potassium loop and enhancing the chlorine loop as known from the cement industry.

Remark: from the above chart, it results that in lime kilns the alkali sulphates will leave the kiln either with the lime or as particulates. Sodium chloride present in the limestone will liquefy in the kiln. A small amount may evaporate in the vicinity of the burner but the overwhelming amount will leave the kiln with the lime pebbles.

3.2.2 Behaviour of potassium feldspar

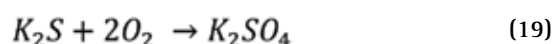
These clays are in general chemically and thermally very stable but they are subject to ion exchange reactions in solid solutions, especially in the presence of sulphate and chloride salts [13] as described in the following equations:



As shown in the previous chapter calcium sulphate and chlorine are always present in lime kilns. Consequently potassium feldspar is a major source of potassium sulphate and chlorine in these kilns through the above reactions.

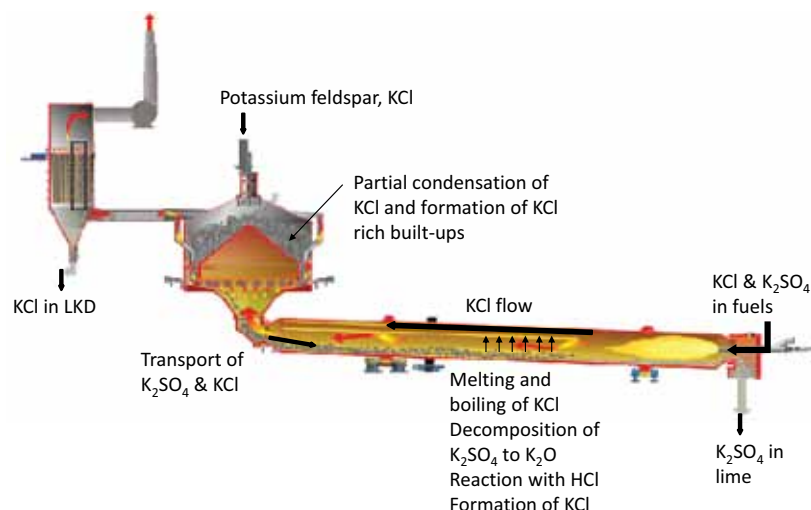
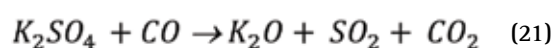
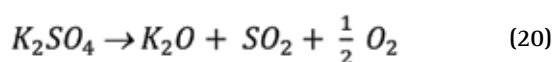
3.2.3 Behaviour of K_2S

During the combustion phase of the fuel, potassium sulphide is converted into potassium sulphate via the following reaction:



3.2.4 Behaviour of K_2SO_4

Similar to calcium sulphate potassium sulphate can decompose either at higher temperatures (> 870°C) and oxidative process conditions or at lower temperatures and reducing conditions as summarised below.



Under oxidising conditions and a residence time of 2 hours at 1000°C, the conversion rate of K_2SO_4 into K_2O is about 15% only, whereas under reducing conditions the reaction rate is much faster and the conversion yield in the order of magnitude of 70%.

The potassium oxide generated in the previous reactions is very reactive with HCl, thus leading to the formation of KCl as described hereafter:



Hence potassium sulphate that is directly fed into the kiln or generated from potassium feldspar or potassium sulphide (according to (17) and (19)) can be finally converted into KCl. The yield of the conversion from K_2SO_4 into KCl depends especially on the oxidative or reducing conditions in the kiln as displayed in Figure 10. It is thus worth underlying that reducing conditions significantly favour the formation of KCl and consequently the formation of build-ups.

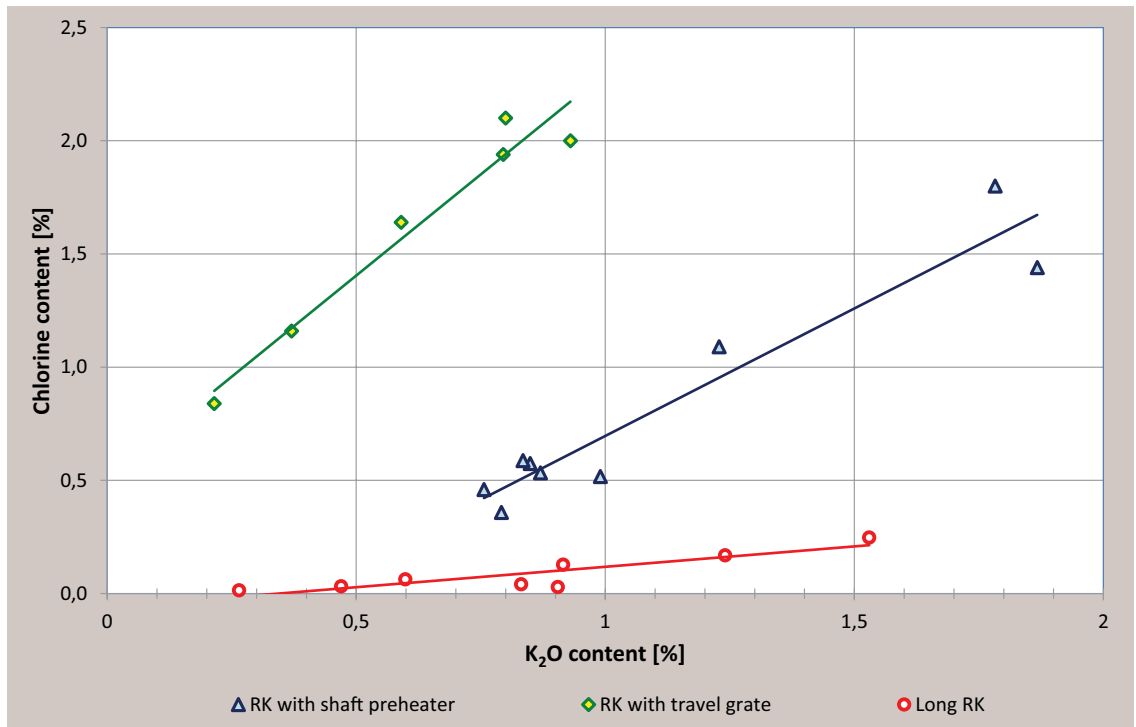
3.3 Fate of potassium

The fate of potassium compounds and the formation of a potassium loop in shaft preheater rotary kilns are displayed in Figure 11. According to different preliminary model calculations and on site measurements it appears that the importance of this loop is a function of some physical characteristics of the preheating zone. Thus rotary kilns can be sorted as follows by decreasing importance of the loops:

- » Kilns equipped with modern shaft preheater
- » Kilns equipped with old shaft preheater
- » Kilns equipped with travel grate preheater
- » Straight or long rotary kilns

11 Potassium chloride loop in a lime rotary kiln with shaft preheater

12 Typical relationship between chloride and potassium contents in the LKD of different types of rotary kilns



According to Figure 11, potassium chloride is collected in the Lime Kiln Dust (LKD) together with other chloride compounds (as shown in Figure 7). This result is based on the following facts:

- » X-ray diffraction (XRD) analyses confirm the presence of KCl in LKD
- » Chemical analyses of LKD show quite often that there is a good correlation ($r^2 > 0,8$) between the chloride and the potassium contents for a given fuel mix and stone quality (see Figure 12), thus suggesting that that chlorine is essentially linked with potassium

Remark: since sodium chloride is the salt with the highest chloride input into the kiln, it can be expected that there would be large amounts of NaCl in the LKD and thus that the chloride and sodium contents would in fact correlate. Since this correlation is clearly not found, the modelling of the chemical reactions that predicts that the chlorine loop is essentially due to a recirculation of potassium chloride is further confirmed.

4 Conclusion

Based on the inventory of the chemical reactions involving sulphur and chlorine in rotary kilns, detailed thermo-chemical calculations could be carried out. In the next step, these enabled a more precise assessment of the fate of both elements in the kilns and in particular:

- » Sulphur and chlorine inputs can lead to the release of SO₂ and HCl in the preheating and the kiln areas. Both gases can be chemisorbed on the external layer of the limestone / lime pebbles in the preheating zone

- » Since the sorbed sulphur and chlorine species are fed back to the kiln, they can be decomposed again leading to the formation of sulphur and chlorine loops

According to the preliminary modelling results, it was found, and frequently confirmed by in-situ measurements, that reducing conditions favour SO₂ emissions and often HCl emissions. However these chemical reactions that considered mainly calcium salts were neither able to predict sufficiently well the magnitude of SO₂ and the HCl emissions of rotary lime kilns, nor the clogging of the preheating zone. Further investigations were consequently performed to increase the accuracy of the model.

These highlighted the major influence of potassium on the HCl emissions and the formation of built-ups in rotary lime kilns. Actually the potassium is introduced into the kiln through the stone feed or the fuels. Following different chemical reactions that were clearly identified and quantified, a substantial amount of this potassium can be transformed into potassium oxide. This substance can then easily react with HCl in the kiln, thus reducing accordingly the HCl emissions but increasing the amount of KCl. As potassium chloride is a volatile salt, it can leave the kiln with the flue gas and then partially condensate in the preheating zone. Part of the potassium chloride goes back to the kiln together with the pebbles thus creating a potassium loop. The part that is captured in the preheater favours the accumulation of dust which leads finally to build-ups that clog the preheater.

Since the chemical reactions occurring in rotary lime kilns are now identified and quantified, the

next step in the modelling will consist of assessing the main process parameters that :

- » Influence the SO₂ and HCl emissions
- » Favour or inhibit the formation of build-ups in preheaters
- » Affect the concentration of sulphur in the lime exiting the kiln, and thus its ability to meet customer requirements

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