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# Investigation of alumina's emission in the deflagration of gas-aluminum particles mixtures

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## Abstract

The objective of this study is the validation and improvement of thermochemical models used in simulations of combustions such as deflagrations and detonations. The validation is done on explosives containing aluminum particles, which oxidizes into alumina during the combustion. One of the objectives is to study the thermochemical states of alumina in the deflagrations of hybrid gas/particle mixtures of H<sub>2</sub>/O<sub>2</sub>/CO<sub>2</sub>/N<sub>2</sub> and aluminum particles. In order to do so, alumina's volume fraction, size and temperature have to be determined in different thermodynamic conditions. These information are obtained by measuring the radiance of alumina by spectroscopic means in the visible and near infrared wavelength regions.

In this work, our aim is to utilize the visible and near infrared ranges where the radiance of alumina is less affected by other parameters such as the emission of the other species. The optimal wavelengths are determined by analyzing the radiance's sensitivity to the searched parameters.

The evolution of the spectra is studied in ranges up to 3600 K and 21 bar, in the visible and near infrared regions. They are compared with the spectra of the same gas mixtures without aluminum particles, all things being equal. This preliminary work will provide information on the optimal wavelength ranges suitable to measure accurately the radiance of alumina, and in further works determine the volume fraction, the temperature and size of alumina particles in deflagrations.

## Nomenclature

$d_p$	Diameter of a particle, $\mu\text{m}$	$T$	Temperature, K
$f_v$	Volume fraction	$P$	Pressure, bar
$K_{\lambda_i}$	Attenuation coefficient of the gas $i$		
$L^0_{\lambda}$	Monochromatic radiance of a black body, $\text{W}\cdot\mu\text{m}^{-1}\cdot\text{m}^{-2}\cdot\text{sr}^{-1}$		<i>Greek symbols</i>
$L_{\lambda}$	Monochromatic radiance, $\text{W}\cdot\mu\text{m}^{-1}\cdot\text{m}^{-2}\cdot\text{sr}^{-1}$	$\lambda$	Wavelength, $\mu\text{m}$
		$\varepsilon_{\lambda}(T)$	Spectral emissivity

## Introduction

The combustion of solid aluminum particles has a great energetic potential. When in contact with oxygen, aluminum reacts and form alumina (Al<sub>2</sub>O<sub>3</sub>). Its use is interesting in energetic materials because it increases the temperature during the explosion. It is however sensitive to many factors, such as the diameter of the particles  $d_p$  and their volume fraction  $f_v$ . The deflagration or detonation of a mixture with aluminum particles also depends on other parameters, such as the composition, the initial pressure and temperature. The study of these phenomenon requires a good knowledge of the effect of these different parameters. Emission spectroscopy is a relevant experimental method to probe the combustion of energetic materials, since they radiate considerably at high temperatures and pressures. For instance, it can give information on the temperature and the composition. Emission lines of chemical species are well-known and modeled at atmospheric pressures and up to a few bars, but there is very little

knowledge for higher pressures starting from 5 bar, as it could occur during deflagrations and detonations.

The aim of this paper is to identify convenient spectral ranges where alumina's emission doesn't interact with those of other species, and where it is sensitive to parameters related to the aluminum particles introduced: their diameter, their volume fraction and their temperature. The investigation is focused in the visible to mid infrared wavelengths (0.3 – 5  $\mu\text{m}$ ), where the radiance of the gases and the particles is high at high pressure and temperature. This knowledge will then be useful in further studies of alumina's emission in deflagration and detonation products of energetic mixtures, for determining its temperature, size and volume fraction.

In order to do so, gaseous deflagrations of  $\text{H}_2/\text{O}_2/\text{N}_2/\text{CO}_2$  with aluminum particles are considered. The main products,  $\text{H}_2\text{O}$ ,  $\text{CO}_2$ ,  $\text{CO}$ ,  $\text{N}_2$ , and aluminum oxide, are the same as those that could be found after the detonation of solid materials. Two mixtures with different compositions of gaseous reactants have been selected, with final deflagration pressures from 8.5 bar to 21 bar, and temperatures from 2 800 K to 3 600 K. The wavelengths ranges are selected according to the following criteria:

- high level of alumina's emission,
- variation of the intensity with the volume fraction, diameter and temperature of aluminum,
- banning the ranges where the mixture's reactants, intermediates and products radiate (other than alumina),
- banning the ranges where chemiluminescence occurs.

The spectra used result from different databases and emission models, as well as experimental measurements by a fast spectroscopic set-up.

## 1. Material and methods

### 1.1. Databases for calculating spectra

The evolution of the radiation's spectrum of a gas mixture can be modelled by spectroscopic databases. For gas mixture at high temperature and high pressure, the HITRAN (2018) (Hill 2016) and HITEMP (2010) (Rothman 2010) are used. They allow to predict and simulate the transmission and emission of gas compounds.

Spectra are then calculated from these databases by different models: HAPI (Kochanov, 2016), and SIAME, a thermochemical code developed at the CEA Gramat (Osmont 2017). Knowing the composition of the reactants, with SIAME, the equations of state can also be predicted, which is helpful for determining the final pressure and temperature of the mixture.

To evaluate the effect of the size and volume fraction of aluminum particles on the radiation, the spectra of alumina is calculated by the method described by Ranc-Darbord, 2018.

### 1.2. The mixtures

Two gaseous mixtures are studied, their final temperature and pressure without aluminum particles have been calculated by SIAME. Their initial and final composition and conditions are detailed in Table 1 and Table 2.

Table 1: Initial composition of the mixtures and their initial temperature and pressure

Mixture	Composition (mole fraction)				$P_i$ (bar)	$T_i$ (K)
	$\text{H}_2$	$\text{O}_2$	$\text{N}_2$	$\text{CO}_2$		
1	0.4054	0.2087	0.1985	0.1873	1.068	298.15
2	0.5358	0.2679	0.1249	0.0714	2.200	298.15

Table 2: Final composition of the mixtures and their calculated temperature and pressure by SIAME

Mixture	Main products (mole fraction)						$P_f$ (bar)	$T_f$ (K)
	$\text{H}_2\text{O}$	$\text{CO}_2$	$\text{CO}$	$\text{O}_2$	$\text{N}_2$	$\text{H}_2$		
1	0.4460	0.1691	0.0542	0.0325	0.2323	0.0208	8.499	2 828
2	0.5567	0.0337	0.0556	0.0729	0.1446	0.1133	21.274	3 612

## 2. Alumina's selected emission ranges

### 2.1. Emission spectrum

The emissivity of the condensed phase of alumina is very low at ambient temperature (Figure 1), so the gas/particle mixture between the radiation of the reacted species and the measuring system can be considered transparent. Therefore, the radiation measured is considered to only come from the products. At a high temperature, alumina is mostly a broad continuous spectrum in the range of 0.300  $\mu\text{m}$  – 5  $\mu\text{m}$  (Figure 1). It varies noticeably between 1.100  $\mu\text{m}$  and 3  $\mu\text{m}$ , if possible this area should be avoided. Alumina's emissivity is greater in the visible range than in the infrared range. The determination of the temperature of a mixture by a spectroscopic method is usually done around 4  $\mu\text{m}$ . Alumina's emissivity is quite high in that range, the radiation measured in these wavelengths should take into accounts alumina's. The sensitivity of alumina's emission to different parameters is studied in the next paragraph, in order to define which spectral zones should be focused on.

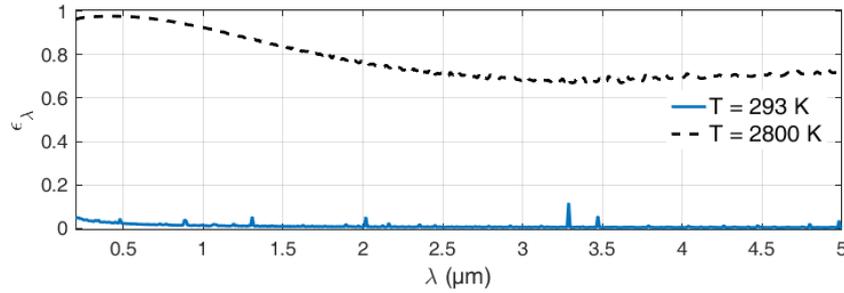


Figure 1: Calculated emission of alumina particles ( $d_p = 25 \mu\text{m}$ ,  $f_v = 1.10^{-4}$ )

### 2.2. The variation of radiance with the particle's temperature, size and volume fraction

The purpose of identifying alumina's spectral ranges is to select the wavelengths where the emissivity of the particle is sensitive to parameters related to its temperature  $T$ , size  $d_p$ , and volume fraction  $f_v$ . They will be useful in further works to determine these same parameters from a radiance measurement. The radiance is modeled using thermophysical calculations in a model developed in Ranc, 2018. The following hypothesis are used:

- aluminum combustion generates large particles and submicron particles;
- the volume fraction is small enough to be in independent scattering conditions (Bouyer 2006), the limits being a diameter of 1  $\mu\text{m}$  minimum and a volume fraction of  $8.10^{-3}$  minimum (Tien 1987);
- the Mie scattering theory is applied to the submicron particles and the diffusion of large particles is neglected.

The radiance is then defined by:

$$L_\lambda(\tau_\ell) = L_\lambda^0(T) \varepsilon_\lambda(\ell, d_p, f_v, T) = L_\lambda^0 (1 - \exp(-\Gamma \tau_\ell) - 2 \frac{r_\infty(r_\infty \exp(-\Gamma \tau_\ell) - 1)}{\exp(\Gamma \tau_\ell) - r_\infty^2 \exp(-\Gamma \tau_\ell)} \sinh(\Gamma \tau_\ell)) \quad (1)$$

with  $\varepsilon_\lambda$  the emissivity of the cloud,  $\ell$  the optical path length,  $\beta$ ,  $\omega$ , and  $g$  computed from de Mie scattering theory,  $\tau_\ell = \beta \times \ell$ ,  $\Gamma = \sqrt{(1 - \omega)(1 - \omega_\lambda g)}$ , and  $r_\infty = \frac{\sqrt{1 - \omega_\lambda g} - \sqrt{1 - \omega_\lambda}}{\sqrt{1 - \omega_\lambda g} + \sqrt{1 - \omega_\lambda}}$ .

The variation of the radiance with the particle's temperature, size and volume fraction is studied by variance-based sensitivity analysis. Here, the Sobol method is applied on the emission calculations from the model (Ranc, 2018) in python using the Salib library. The approach consists in determining Sobol indexes from the decomposition of the variance of the emissivity. The first order index,  $S_1$ , represents the variation of the emission related to each parameter, independently. The total effect index,  $S_i$ , is the variation of each parameter taking into account their interaction with the other parameters. Finally, the interaction index defines the interaction of two parameters; the lower it is, the more independently the parameters influence the emissivity. The results obtained are presented in Figure 2.

The effect of the temperature on the emissivity in the VIS/NIR range is low, its first order index  $S_1$  is under 0.1 until 1.500  $\mu\text{m}$ . The temperature has a very small impact on the emissivity (nearly none in the

visible range), except in the infrared region between 1.500  $\mu\text{m}$  and 3.500  $\mu\text{m}$ . The interaction effect index of all 3 parameters is below 2%, which explains why the total indexes have nearly the same value as the first order indexes for each parameter. So they all influence the variation of the emissivity independently.

The total effect index  $S_T$  according to the volume fraction is quite high: it is around 0.8 in the IR range (1.2  $\mu\text{m}$  – 5 $\mu\text{m}$ ), and between 0.6 and 0.77 in the VIS/NIR range, therefore the emission of alumina is very sensitive to the volume fraction. The total effect index  $S_T$  according to the diameter of the particles is below 2 in the IR range (1  $\mu\text{m}$  – 5  $\mu\text{m}$ ) and rises up to 0.38 in the VIS/NIR range. The emission is more sensitive to the diameter in that range.

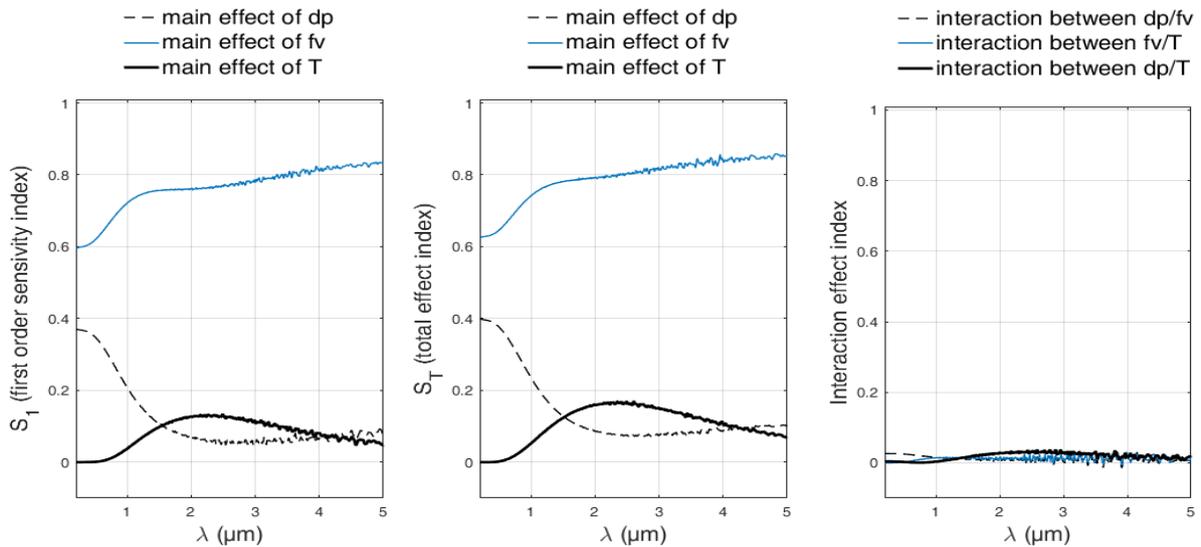


Figure 2: Sobol' indexes for the variance of the emissivity of an alumina cloud for particle diameters from 25 to 100  $\mu\text{m}$ , volume fractions from  $1 \cdot 10^{-3}$  to  $1 \cdot 10^{-6}$  and temperatures from 2500 to 3600 K

To illustrate these results, the variation of the emissivity when the volume fraction changes is showed in Figure 3 for two constant particle sizes, at 2750 K. The emissivity varies greatly with the volume fraction, like it was expected. The variation of the radiance when the size of the particle changes is showed in Figure 4 for two constant volume fractions, at 2750 K. The smaller the volume fraction is, the lower the emissivity is. The NIR range seems suitable for studying the effect of the diameter, the emissivity being too similar in the visible range for volume fractions around  $1 \cdot 10^{-3}$ , and also in the infrared range for small volume fractions. The variation of the emission when the temperature of the particle changes is showed in Figure 5, for two volume fractions. The emissivity varies greatly in the infrared range, but barely in the visible range, and there are nearly no differences between the curves at 3100 K and 3600 K.

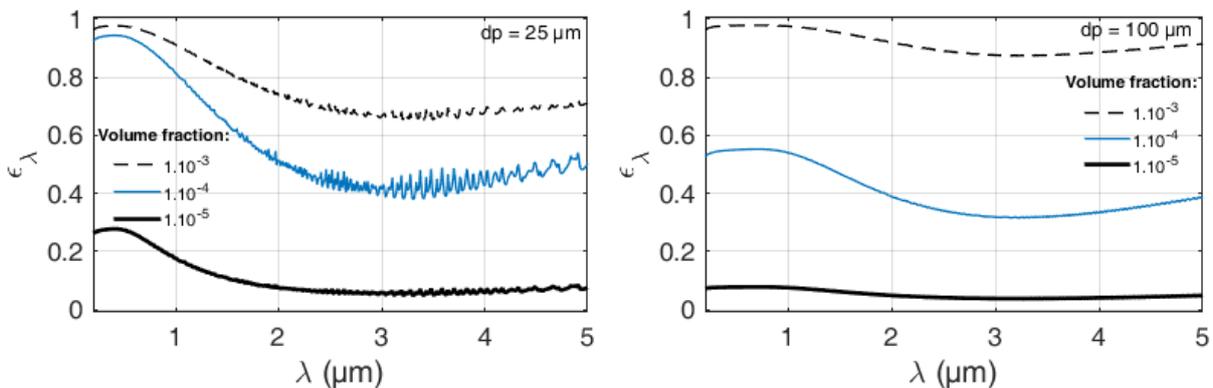


Figure 3: Variation of emissivity of alumina with volume fraction, at  $T = 2750$  K

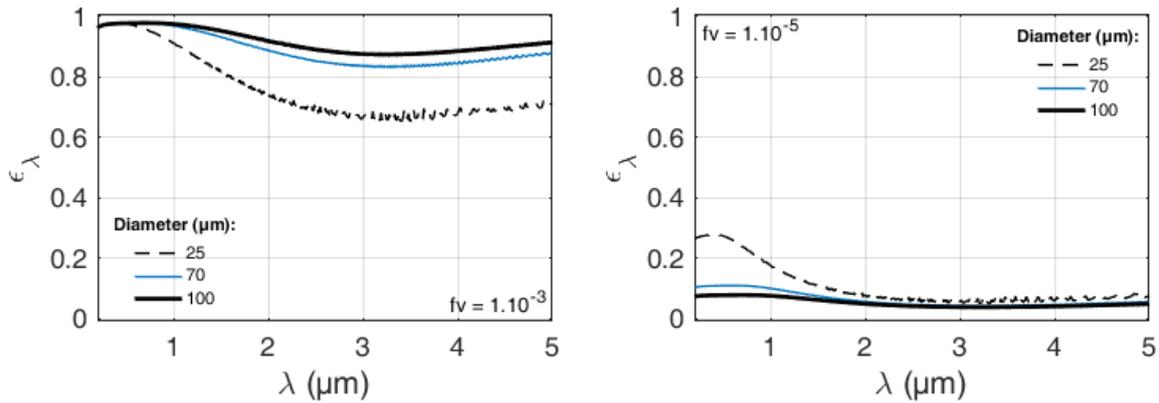


Figure 4: Variation of emissivity of alumina with particle size, at  $T = 2750\text{ K}$

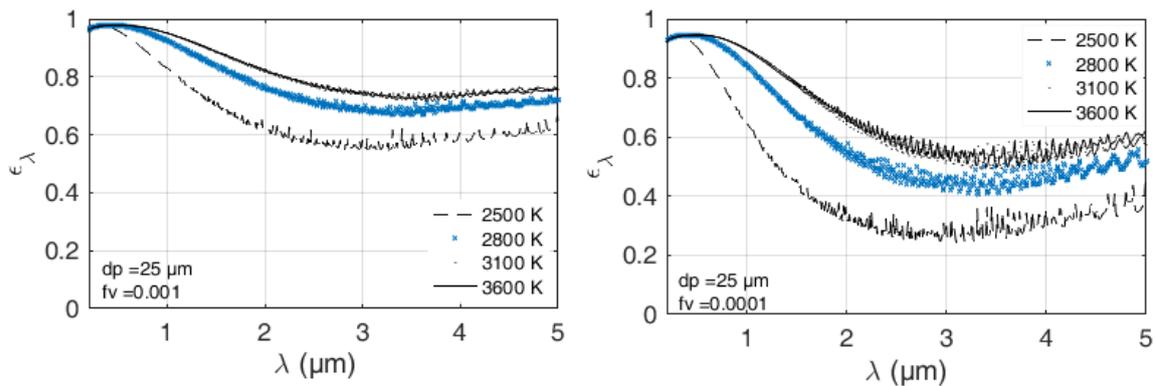


Figure 5: Variation of emissivity of alumina with temperature

### 2.3. Summary of selected alumina's wavelength

The emissivity of alumina is mostly sensitive to the volume fraction, but it is also quite much to the particle diameter, especially in the VIS/NIR range. These two parameters could be studied between  $0.300\ \mu\text{m}$  and  $1.300\ \mu\text{m}$ . However, for the higher volume fractions ( $> 1.10^{-3}$ ), the variation of emissivity is lower. It would then be better to consider the NIR range ( $0.700\ \mu\text{m}$  to  $1.100\ \mu\text{m}$ ).

The emissivity varies little with the change in temperature in the VIS/NIR range, it is best to study it between  $1.500\ \mu\text{m}$  and  $3.500\ \mu\text{m}$ . In experimental measurements, the measured radiance  $L_\lambda(T)$  is the product of the emissivity  $\epsilon_\lambda(T)$  and the radiance of a black body  $L^0_\lambda(T)$ , at each wavelength. The temperature can then be determined using Planck's law, which relates the radiance of a black body to its temperature.

## 3. Wavelengths to avoid

### 3.1. Emission from the gaseous deflagration without aluminum

First, the chemical species from the mixture without Al is considered to identify the main gaseous species that radiate. The mechanism of the  $\text{H}_2/\text{O}_2$  reaction involves many steps (Maas 1988). The main radiating species are the following:

- the reactants:  $\text{H}_2$ ,  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{CO}_2$ ;
- the intermediates:  $\text{CO}$ ,  $\text{O}$ ,  $\text{H}$ ,  $\text{NO}$ ,  $\text{OH}$ ,  $\text{H}_2\text{O}$ ,  $\text{H}_2\text{O}_2$ ;
- the main products (other than the reactants that didn't react):  $\text{H}_2\text{O}$ .

The overall spectrum calculated for the deflagration of the two mixtures presented in 1.2 is given in Figure 6. The calculations are done using the final composition, temperature and temperature in Table 2. There is not any emission in the visible region, until  $0.800\ \mu\text{m}$ . Water radiates from  $0.800\ \mu\text{m}$  to  $0.900\ \mu\text{m}$ , but its intensity is so low it can be neglected. The gases mainly radiate in the infrared region. In the NIR, there are a few  $\text{H}_2\text{O}$  emission lines to avoid, at  $0.900\ \mu\text{m}$  to  $1.000\ \mu\text{m}$  and at  $1.080\ \mu\text{m}$  to  $1.200\ \mu\text{m}$ .

The range with no other emission lines is between 0.800  $\mu\text{m}$  and 0.885  $\mu\text{m}$ . In the infrared region, gases radiate greatly, there is only a small range that could be used, between 2.100  $\mu\text{m}$  and 2.200  $\mu\text{m}$ , if the few low emission lines distinguishable are neglected.

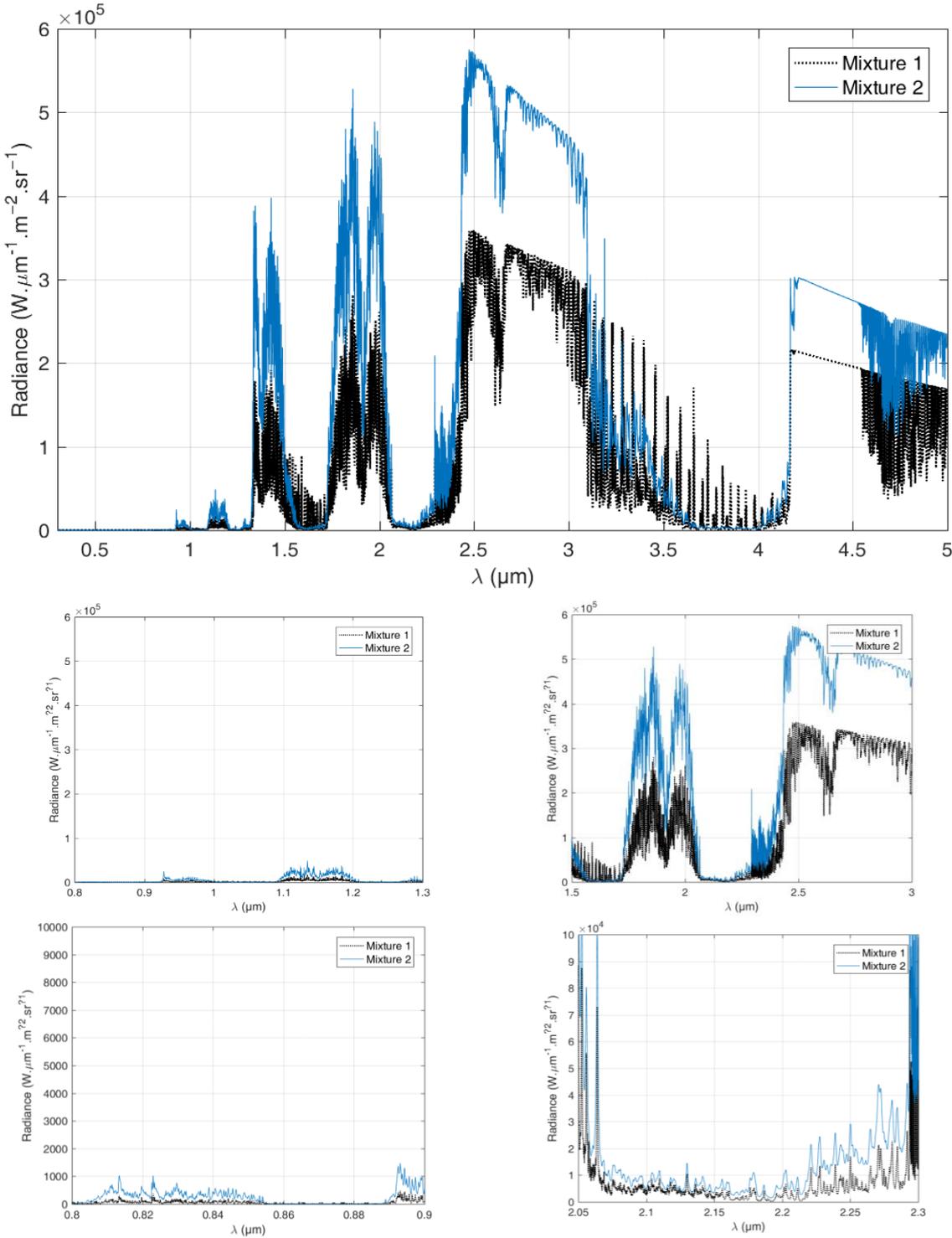


Figure 6: Calculated spectra of the deflagrations of the mixtures without aluminum

### 3.2. Emission from aluminum’s reaction during the deflagration

In this paragraph, the reaction of aluminum during the deflagration is considered. Aluminum mainly reacts with oxygen, the mechanism is presented in Table 3 (Bocanegra 2008). The overall equation is given by (2). The reactions essentially occur in gaseous phase: first the mixture heats up, then the solid

aluminum particles melt into a liquid phase (reaction #1) and the remaining reactions occur. Since aluminum and alumina change into a liquid phase, a saturation vapor appears. A thermodynamic equilibrium between the liquid and the gaseous phases occurs at each pressure.



The main radiating species (other than does cited in 3.1) are the following (Sarou-Kanian 2003):

- the emission of the aluminum that didn't react;
- the emission of the main intermediates: AlO, Al<sub>2</sub>O, AlO<sub>2</sub>.

The main spectral lines of each molecule are reported in Table 4. They radiate exclusively in the visible range from 0.300 μm to 0.500 μm.

*Table 3: Aluminum oxidation mechanism (Bocanegra 2008)*

#	Reaction
1	Al=Al <sub>(SL)</sub>
2	Al <sub>2</sub> O <sub>3</sub> =Al <sub>2</sub> O <sub>3(L)</sub>
3	Al <sub>2</sub> O <sub>3(L)</sub> =Al <sub>2</sub> O <sub>3(S)</sub>
4	Al+O <sub>2</sub> =AlO+O
5	Al+O+M=AlO+M
6	AlO+O <sub>2</sub> =OAlO+O
7	Al <sub>2</sub> O <sub>3</sub> =AlOAlO+O
8	Al <sub>2</sub> O <sub>3</sub> =OAlO+AlO
9	AlOAlO=2AlO
10	AlOAlO=Al+OAlO
11	AlOAlO=AlOAl+O
12	OAlO=AlO+O
13	AlOAl=AlO+Al

*Table 4: Main spectral lines of aluminum compounds during the formation of alumina*

Species	Main spectral lines (μm)
Al	0.3092 - 0.3944 - 0.3961
AlO	0.464 μm 0.486 μm 0.507
Al <sub>2</sub> O	0.429
AlO <sub>2</sub>	0.500

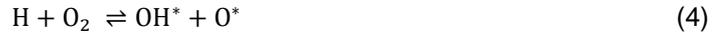
### 3.3. Non thermic emissions from chemiluminescence

Chemiluminescence can occur during the chemical oxidation in combustions, where molecules are yielded to electronically excited states and form radicals (denoted \*). It can be caused by quenching (denoted M), when a molecule or a photon gains energy during a collision. When these molecules relax from the excited state to their ground state, a photon is released. It should be mentioned that there is not always a consensus on the mechanisms of such chemical reactions, they are not always well known. The emission produced usually consists of a broadband continuum difficult to isolate. Its intensity is proportionate to the concentration of the reacting molecule. In this study, the main chemiluminescent species are CO<sub>2</sub>\*, OH\*, and AlO\*.

A reactional mechanism leading to the chemiluminescence of CO<sub>2</sub>\* is given in Kopp, 2012. The main reaction producing CO<sub>2</sub>\* is expressed in (3), and causes a broad continuum mainly in the visible range between 0.300 and 0.650 μm (Kopp 2012, Bozkurt 2013, Ding 2018). At temperatures above 1000 K, the radiation becomes more intense.



The main reaction yielding OH\* at high temperature and low pressure is given by (4), which becomes competitive with a pressure and temperature dependent reaction given by (5) (Kathrotia 2011, Bozkurt 2013). Above 2800 K, OH\* is also formed by thermal excitation, independent from (5) (Hidaka 1982, Koike 1982). The chemiluminescence of OH\* is mainly a peak at 0.309  $\mu\text{m}$  (Haber 2003, Kopp 2012, Ding 2018)



The mechanism of AlO\* chemiluminescence is not well known. To stay consistent with the reaction of Al to Al<sub>2</sub>O<sub>3</sub> mechanism chosen in this study (Bocanegra 2008), the electronic quenching reaction #5 described by (6) is chosen (Ribière 2008). According to the emission of AlO\* detailed in other studies (Kolb 1975, Oblath 1980), there is an emission of a broad continuum between 0.500 and 0.640  $\mu\text{m}$ , and another starting at 400 nm fading between 700 and 800 nm. However, the intensity of AlO\* chemiluminescence might be quite weak with O<sub>2</sub> as the oxidant (Rosenwaks 1975).



Although chemiluminescence in combustions in the VIS/NIR range is quite much investigated, there are scarcely any studies in the infrared range (Pettersson 2004). No bands are mentioned here, but chemiluminescence can occur as well in these ranges (Dabos 2019).

### 3.4. Summary of the wavelengths to avoid

The wavelengths to avoid are summed up in Table 5.

Table 5: Summary of the wavelengths to avoid

Wavelength(s) ( $\mu\text{m}$ )	Origin
0.300 – 0.650 <i>continuum</i>	Chemiluminescence of CO <sub>2</sub> *
0.309 <i>peak</i>	Chemiluminescence of OH*
0.500 – 0.640 <i>continuum</i>	Chemiluminescence of AlO*
0.400 – 0.800 <i>continuum</i>	
0.3092 – 0.3944 – 0.3961 <i>peaks</i>	Al emission
0.464 – 0.486 – 0.507 <i>peaks</i>	AlO emission
0.429 <i>peak</i>	Al <sub>2</sub> O
0.500 <i>peak</i>	AlO <sub>2</sub>
0.800 – 1.000 <i>peaks</i>	H <sub>2</sub> O emission
1.080 – 1.200 <i>peaks</i>	
1.200 – 2.100 <i>peaks</i>	
2.200 – 5.000 <i>peaks</i>	Gas mixture emission

## 4. Selecting the ranges of wavelengths

Given the wavelengths to avoid in Table 4, and the determination of the ranges where the variation of the three targeted parameters (particle diameter, volume fraction and temperature), two different ranges can be defined for studying the emissivity of alumina. The first one, between 0.800  $\mu\text{m}$  and 0.885  $\mu\text{m}$ , is interesting for studying the particle's diameter and the volume fraction. The second one, in the infrared wavelengths between 2.100  $\mu\text{m}$  and 2.200  $\mu\text{m}$ , is relevant for investigating the temperature of alumina. The temperature of the gases is determined using Planck's law, which defines the relation between the radiance and the temperature of a black body at all wavelengths. At high temperature and high pressure, CO<sub>2</sub> behaves like a black body between 4.17  $\mu\text{m}$  and 4.19  $\mu\text{m}$  (Fournet 1997, Ramel 2008), the radiance measured in this range allows the use of Planck's law.

## Conclusion

This study contributed to highlight the relevant wavelengths ranges where the emissivity of alumina produced from gas-aluminum particles mixtures is sensitive to the particle's diameter, volume fraction and temperature. In summary, with the gases that radiate greatly in the infrared wavelengths, the aluminum compounds that radiate in the visible range and the chemiluminescent emissions, the ranges available are quite narrow. A variance-based sensitivity analysis helped ensuring these ranges were relevant for studying the emission of alumina with the variation of the three parameters mentioned. These ranges can then be used in further works to determine these parameters from the measurement of the radiation of a deflagration. These data will then be useful for validating thermochemical models, especially at high pressures and temperatures where there are less valid modeling of the emission lines (Ranc 2018). Besides, chemiluminescence occurring in combustions in the infrared range is not well known, it would require further investigation, especially for  $\text{AlO}^*$ .

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