STUDY, DESIGN, IMPLEMENTATION AND EFFICIENCY OF ATMOSPHERIC EMISSION CLEANING SYSTEMS IN THE CERAMIC FRIT MANUFACTURING PROCESS

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ABSTRACT

A study has been undertaken of a gaseous emission cleaning system based on the use of baghouse filters, installed at QUIMICER, S.A., for treatment of the emissions from two frit melting kilns.

The application of such cleaning systems in filtering this gas stream faces a particular difficulty, owing to the nature of the solid particles present, since semi-molten particles are involved, which can condense on the surface of ducts, heat exchangers, baghouses, etc. This parameter has thus been one of the most important design parameters of the treatment facility. On the other hand, the studied facility features the singularity that frit melting kilns operate using oxygen as an oxidising agent (oxycombustion).

Treatment plant design has envisaged integrated eco-management: the waste materials collected in the baghouses are valorised as raw materials by introducing them in the same frit manufacturing process, so that the emission treatment system generates no further wastes.

The following organisations have collaborated in the study: QUIMICER, S.A., at whose facilities the work was conducted; the firm ATECMA of GRUPO G. y C., which designed, installed and put the cleaning system into commission; and Instituto de Tecnología Cerámica (ITC), which characterised the gaseous emissions, waste material collected in the baghouse filters, and has collaborated in control and monitoring of the filtration system and frit melting kiln operating parameters.

1. INTRODUCTION

The main environmental issue associated with frit production is gaseous emission into the atmosphere. The gas emission contains particulates – as a result of solid or semi-molten particles being swept along by the gases, or sublimation of volatilisations when gas and pollutant temperatures decrease in the gaseous stage[1-5].

In the present study, a solid particulate cleaning system has been used for each kiln. The treated material is recovered in the same process by the installation of a pneumatic transport system to the silo that feeds each kiln. The system has been designed in such a way, that the effect of the cleaning system on kiln operation is very low. The two baghouse filters connected to the kilns have a single emission source into the atmosphere, thus simplifying control tasks and controlling systems.

In the present facility, a particularly important operating characteristic with regard to the final results is the use of oxygen as an oxidising agent in the combustion process. The advantages of this oxidising agent include an increase in flame temperature, decrease of the final gas flow to be treated, and therefore reduction of the dimensions of the cleaning system.

2. DESCRIPTION OF THE CLEANING SYSTEM

The experimental work described in this study has been conducted at the facility schematically illustrated in Figure 1. At this facility, the baghouse filter was connected to two frit melting kilns that were making two of the most representative frits currently marketed.

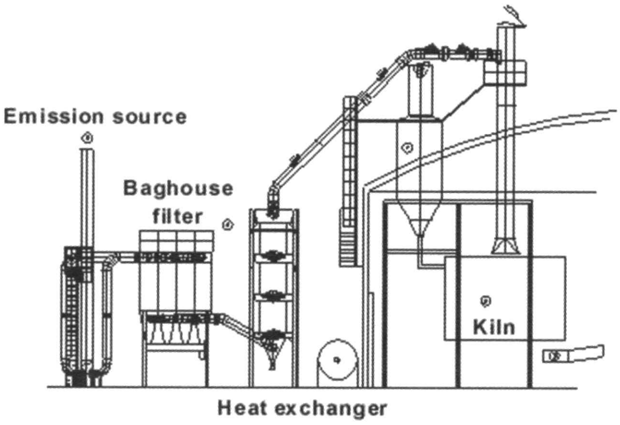

Figure 1. Basic scheme of the studied industrial facility.

The details of the facility outlined in Figure 1 are briefly explained below.

In general terms, the studied treatment facility consists of one heat exchanger per kiln, which cools the gaseous stream to a temperature below 180°C, one baghouse filter per kiln in which the solid particles are separated from the gaseous stream, and an exhaust stack common to the two kilns for release of the cleaned gases into the atmosphere.

Figures 2 and 3 display images of the layout of the cooling (heat exchanger) equipment and filtration facility. The parts of the cleaning system, from the kiln output to the release of the treated emission stream, are set out below.

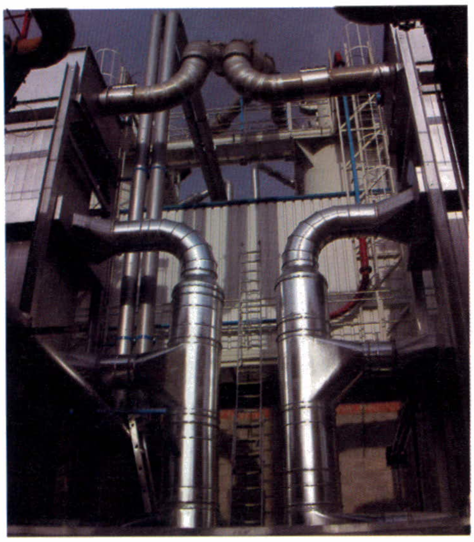

Figure 2.- Cooling equipment layout

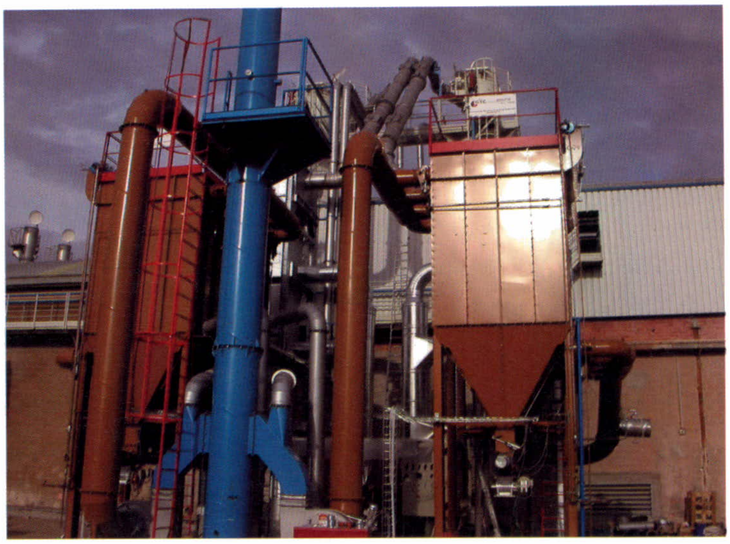

Figure 3.- General view of the filtration facility

A stainless steel structure that served as a by-pass was fixed in the final part of the kiln stack, diverting the flue gases through a suitably dimensioned stainless steel tube to the exchanger. A cleaned air inlet was made in the top part of the stack to reduce the gas stream temperature. That is, a part of the air cleaned in the filter was recirculated through a duct to the top of the stack to cool the flue gases from about 600°C to a maximum temperature of 400°C, which is the required flue gas temperature when the gases enter the heat exchanger.

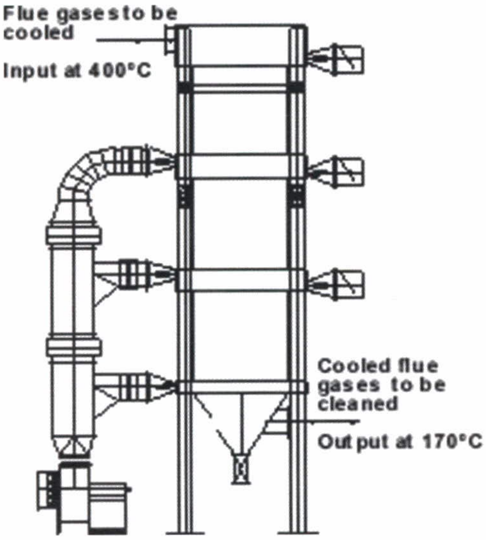

Figure 4. Schematic illustration of heat exchanger parts

Figure 4 schematically illustrates the heat exchanger used in the facility. The exchanger is of the tube bank type, with a tranquillisation chamber, input plenum and dust collection hopper. It has a tube cleaning system by means of pressurised air injection in the flue gas circulation direction. The cleaning sequence is commanded by a pressure differential measurer (ΔP) between the exchanger output and input.

The body of the heat exchanger has three cooling stages. Each stage consists of a tube bank through which the flue gases travel, and countercurrent circulating cooling air in contact with the tube outer surface along its entire perimeter for heat exchange. Several cooling stages were designed to appropriately direct the cooling air, and thus enhance heat exchange efficiency.

A centrifugal fan provides the necessary air flow at ambient temperature to cool the flue gases circulating inside the tube bank from 400ºC to 170ºC. Each of the three stages that make up the body of the exchanger is thus independently cooled by air from this fan through a galvanised tube fitted with three intakes.

There is furthermore a flue gas temperature control system at the exchanger output with a valve for false air intake in case the temperature is high (>180ºC), and three butterfly valves at the output of the three cooling stages to regulate the flow rate.

The baghouse filter is of the modular cell-type, with a rectangular cross section, and the tefloned Nomex filter bags are cleaned by a compressed air injection system (Figure 5). At each cleaning, the dust retained in the bags falls onto an endless screw that conveys the dust to a storage container for subsequent pneumatic transport to a buffer silo that feeds the kiln. The cleaning routine is commanded by a pressure differential measurer (ΔP) between the filter output and input.

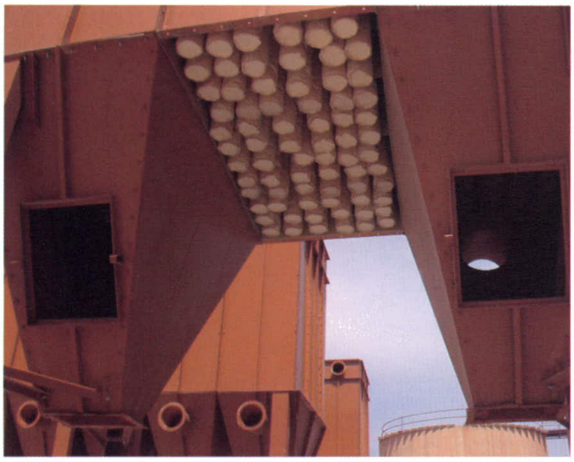

Figure 5. Detail of the bags

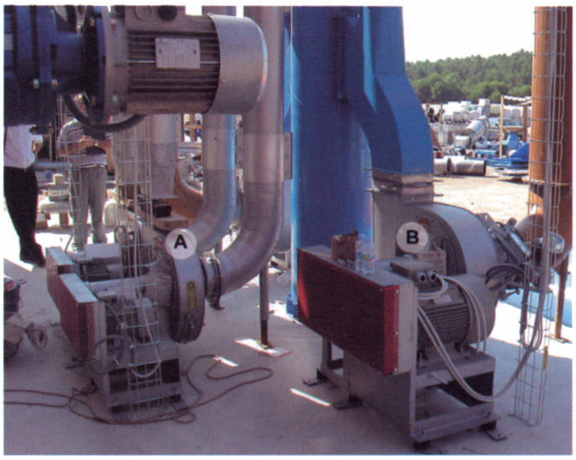

Figure 6. Detail of the fans

Figure 6 shows the fans in the final part of the filtration facility. One of the main novelties in this cleaning system design lies in the cleaned air recirculation by means of the fan at point A. This centrifugal fan provides the necessary air flow by means of modulating valves commanded by the signals received respectively by the pressure transducers and temperature probe, in order to maintain the pressure and temperature values set in the thermoregulator (T_{max} = 400ºC).

The fan that regulates charge loss in the facility and drives the final emission is located at point B. A measuring point is located before the exchanger, which records pressure on-line and regulates the flow extracted by the final fan of the facility (Point B), with a view to regulating pressure in the kiln (draw). In order to keep a constant kiln draw, an automatic Dapo type radial blade valve is actuated, installed in the suction opening of this fan.

The facility is automatically controlled by an independent electric programming for each cleaning system, connected to a differential pressure recording system

(Figure 7) that saves the history of the continuous filtration and bag cleaning process, recording the system charge losses on-line.

Figure 7. Detail of the on-line charge loss recording system (ΔP).

In order to avoid condensates in the filtration system, it is important that the baghouses and ducts should be heated. This system encourages the proper working of the facility during operation, and especially after shutdowns and start-ups for maintenance.

3. OBJECTIVES

- I. Design, implementation and fine-tuning of a facility for particulate removal from frit melting kiln emissions based on baghouse filters.
- II. Study of the operation of the installed cleaning facility, quantifying and characterising the arising solid wastes, as well as the emissions generated in the kilns before and after the filtration stage.
- III. Analysis of treatment facility efficiency and comparison of the results with the existing regulatory limits in matters of particulate removal.

4. EXPERIMENTAL DEVELOPMENT

4.1. MATERIALS FABRICATED

In the tests, the two kilns connected to the cleaning system were fabricating a frit used for obtaining transparent glazes (FT) in single-fired tile manufacture, and a frit used for obtaining opaque glazes (FO) by the presence of zirconium oxide, which forms zirconium silicate crystals that opacify the glaze.

Table 1 details the compositional ranges of the two types of frits involved. In view of their intended use in the manufacture of single-fired porous tiles, both frits had a relatively high proportion of alkaline-earth oxides in their composition to raise the glaze sealing temperature and facilitate outgassing of the CO_2 evolving from the carbonate decomposition reaction in the porous body during the single-fire ceramic tile manufacturing process.

TYPE OF FRIT	SiO_2 (%)	Al_2O_3 (%)	B_2O_3 (%)	CaO (%)	Na_2O (%)	K_2O (%)	ZnO (%)	MgO (%)	ZrO_2 (%)	BaO (%)
FO	55-60	4-7	3-6	9-11	0-1	3-5	8-11	2-4	6-8	-
FT	50-60	4-7	2-5	8-10	0-1	2-4	8-11	1-3	-	0-2

Table 1. Typical oxide composition of the types of frits being made (% by weight)

4.2. MONITORING OF KILN OPERATING PARAMETERS

During the performance of the tests, samples were taken from the processed materials and kiln operating parameters were monitored by means of the control equipment installed in the kilns, complemented by the manual measurement of certain additional parameters. Specifically, we monitored the fuel and oxidising agent (oxygen) flow rate, kiln temperature and pressure, gas temperature in the output collector, production, and mass yield, estimating the time of residence of the material in the kiln.

4.3. CHARACTERISATION OF GAS EMISSIONS

The sampling points for gaseous stream characterisation were positioned:

- **Before the filter:** The point was located in the duct between the heat exchanger and the baghouse filter (Figure 8). In this case there was one sampling point for each baghouse, i.e., per kiln.
- **After the filter:** There was a single sampling point in the emission stack, in which the two treated streams from the baghouses were both mixed (Figure 9).

Figure 8. Gas sampling point before the filter.

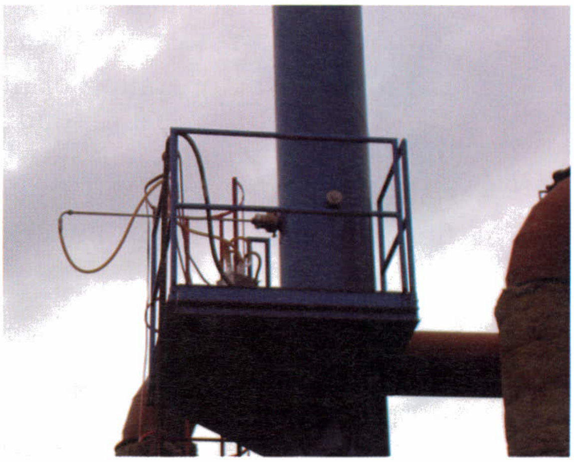

Figure 9. Gas sampling point after the filter.

The methodology used to determine the solid particles present in a gaseous stream is based on the extraction of a certain known volume of this stream and putting this through a particle-retaining filter. After determining the retained particulate quantity by weighing, the particle concentration in the gaseous stream is then calculated.

The test method used to determine solid particulate concentration in the gaseous stream is based on standard UNE 77223:1997[6]. In these conditions, according to the standard, the solid particulate concentration can be established with a margin of uncertainty of ±10%. At ITC, a measurement methodology has been developed adapted to the ceramic industries, which simplifies in certain points the specifications of standard UNE 77223:1997, based on the results obtained during the development of a study in this respect[7].

ITC is accredited by the Spanish National Accreditation Body (ENAC) for performing the determination of solid particulate concentrations in the gaseous stream according to this internal procedure adapted to the ceramic industries.

In the present study, all the measurements were conducted according to the adapted procedure.

4.4. CHARACTERISATION OF THE WASTE HELD IN THE BAGHOUSE FILTERS

The solid material obtained by filtration of the gaseous stream, after the baghouse cleaning stages, was collected separately from each filter. This waste was quantified by weighing, and samples were taken for characterisation.

The material obtained was subjected to the following determinations:

- Chemical analysis: Boron was determined by potentiometric titration, using NaOH 0.1N as a titrant. For the fluorine determination, the sample was disaggregated by alkaline fusion and fluorine was determined by potentiometry with a fluorine selective electrode. The determination of the rest of the elements was performed by X-ray fluorescence spectrometry.
- Mineralogical analysis: For the qualitative determination of the mineral species present in the wastes, an X-ray diffraction analysis instrument was used. Complementing this analysis by chemical analysis allowed estimating the emission of materials in the crystalline phase and the glassy phase.
- Observation by scanning electron microscopy (SEM): The waste samples were observed, photographed and analysed with an energy-dispersive X-ray microanalysis (EDXA) instrument connected to a scanning electron microscope.
- Particle-size distribution (PSD): PSD was determined using a laser diffraction instrument. In this instrument, the particles cross a beam of laser light and the resulting scattered light is collected in an array of 40 detectors. Size distribution was calculated by the instrument's software, using the Fraunhofer diffraction model to interpret the scattered light signal. The dust is fed into the instrument, applying a vacuum and injecting air, and crosses a system of ducts at high speed, where the impacts and shear stresses break up the agglomerates and individualise the particles.

5. RESULTS AND DISCUSSION

5.1. FRIT MELTING KILN OPERATING PARAMETERS

The values of the characteristic kiln operating parameters in place during the tests, while the above frit compositions were being fabricated, are given in Table 2. These values remained practically unchanged during the experimental study.

		KILN 6	KILN 7
Type of frit made		FT	FO
Combustion	**Mean natural gas flow rate (m^3/h)**	180	180
	Mean oxygen flow rate (m^3/h)	360	360
Operating conditions	**$t_{material\ residence}$ (min)**	≈90	≈90
	P_{kiln} (mmwc)	1	2
	T_{kiln} (°C)	1440	1470
	$T_{kiln\ flue\ gases}$ (°C)	1140	1110

Table 2. Kiln operating parameters during the tests

During the performance of the tests it was furthermore verified that the quality of the frits made was not altered by the fact that the kiln was running while connected in series to a flue gas cleaning system. This was confirmed during the course of the tests by sampling the resultant frits and subjecting these samples to control tests, which showed no significant changes with regard to the set target values.

5.2. FILTER WASTE CHARACTERISATION

The samples taken from each filter were respectively referenced RFT (waste from the kiln making frit FT) and RFO (waste from the kiln making frit FO).

5.2.1. Chemical and mineralogical analysis

Table 3 sets out the chemical analysis data obtained of waste samples RFT and RFO.

	SiO_2	Al_2O_3	B_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	ZrO_2	PbO	ZnO	As_2O_3	F	Cl	S	LOI
RFT	22.3	4.6	17.8	0.14	9.6	0.99	0.25	11.0	<0.02	0.20	29.2	<0.01	0.27	2.7	0.64	3.78
RFO	26.5	3.6	16.6	0.07	7.84	1.98	0.75	6.64	4.05	0.06	30.4	<0.01	0.1	2.0	0.17	1.50

Table 3. Composition in oxides of the RFT and RFO waste samples (% by weight)

These waste samples were subjected to X-ray diffraction analysis to determine the mineralogical species present. The resulting diffractograms are shown in Figures 10 and 11. Table 4 details the principal mineral species found.

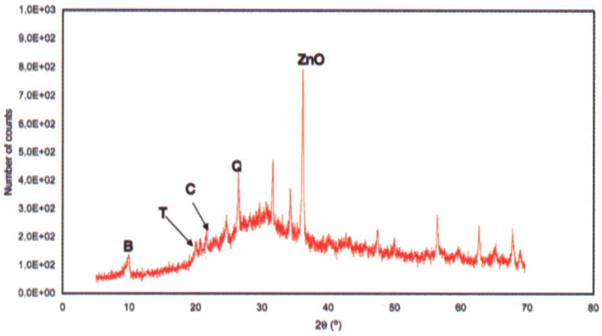

Figure 10. Mineralogical analysis. Sample RFT

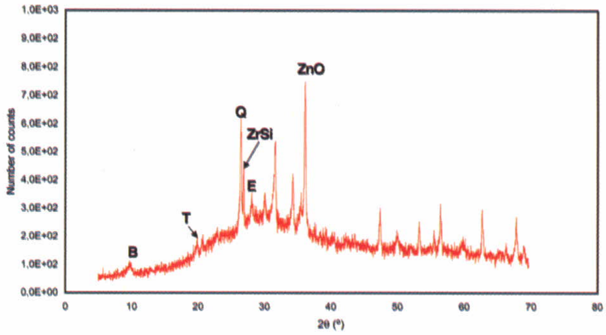

Figure 11. Mineralogical analysis. Sample RFO

Sample	Reference	Mineralogical species (arranged in decreasing order of content)
RFT	**ZnO** **Q** **B** **C** **T**	Zinc oxide (ZnO) Quartz (SiO_2) Sodium-calcium borate ($NaCaB_5O_8(OH)_2\ 3H_2O$) Cristobalite (SiO_2) Tridymite (SiO_2)
RFO	**ZnO** **Q** **ZrSi** **B** **E** **T**	Zinc oxide (ZnO) Quartz (SiO_2) Zirconium silicate ($ZrSiO_4$) Sodium-calcium borate ($NaCaB_5O_8(OH)_2\ 3H_2O$) Enstatite ($Mg_2Si_2O_6$) Tridymite (SiO_2)

Table 4. Results of the qualitative mineralogical analysis.

The results obtained allow making the following inferences:

- The chemical composition of the wastes from the filters, compared with the frit composition, displays a smaller content in more refractory compounds (silica, alumina or zirconium). On the other hand, the composition has a larger content in more fluxing constituents (boron and alkaline), compounds that have probably been released by volatilisation from the melt. It further exhibits zinc oxide enrichment. This compound must essentially be swept along by the kiln gases, because it is introduced as a raw material with a small particle size (mean diameter medium of 1-2 μm), and its thermal behaviour does not justify its presence in such a high percentage in the resulting waste. This assumption is confirmed in the mineralogical analysis data, in which an important zinc oxide content is detected as a crystalline species in both studied wastes.
- A high boron content is observed in the two studied wastes (of the order of 17% B_2O_3). A part of this is present in the wastes in a crystalline phase (like sodium-calcium borate), as shown in Table 4. This indicates that a boron compound fraction is emitted on being swept along by combustion gases.
- The losses on ignition at 900°C (LOI) of the wastes are relatively high. It is to be noted in this sense, that the wastes contain unstable amorphous phases. These can be carbonated or hydrated when in contact with a combustion gas stream that has a high water and CO_2 content. The waste also contains crystalline species of the calcite or borate type, which display losses on ignition at 900°C.
- The diffractograms indicate an important presence of glassy phase, since the diagrams clearly show a glassy halo in the two samples. According to the literature[8], these glassy phases basically correspond to particles formed by a volatilisation process from the melt with subsequent condensation during cooling of the gaseous stream. This interpretation is consistent with the results obtained, when the results of the chemical and mineralogical analysis are compared.
- It is further to be noted that the wastes arising during the tests contained very low quantities of toxic compounds, such as lead or arsenic, because the content of these impurities has been limited in the raw materials used.

The chemical and mineralogical analysis data indicate that the waste has an important economic value (0.2-0.3 €/kg). For this reason, from an economic and environmental point of view, it would be very interesting to be able to recycle the waste as a raw material in the same manufacturing process.

5.2.2. *Particle-size distribution (PSD) analysis*

Figures 12 and 13 plot the PSD obtained by a dry method from the wastes collected in the filters. Hardly any significant differences are observed between both PSD: mean diameter (d_{50} in % by volume) lies at a value of 12.26 μm for waste RFT and 11.65 μm for waste RFO. Both wastes display a relatively wide PSD, with 10 vol% particles larger than 50 μm, and a similar particle percentage smaller than 2.5 μm.

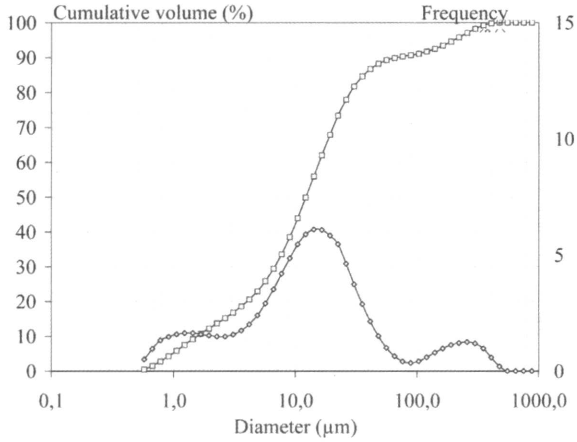

Figure 12. PSD: RFT waste

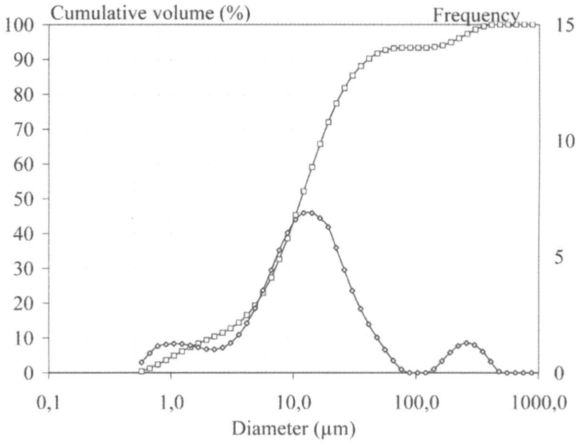

Figure 13. DTP: RFO waste

5.2.3. *Observation by scanning electron microscopy (SEM)*

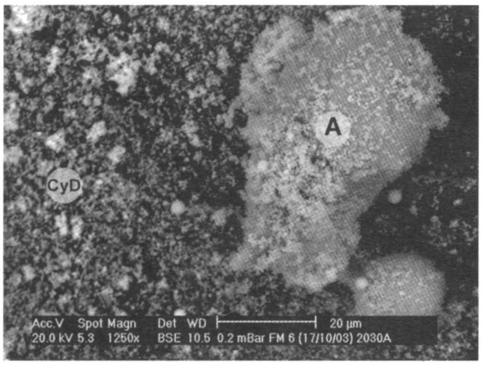

Figure 14. SEM photographs: waste RFT

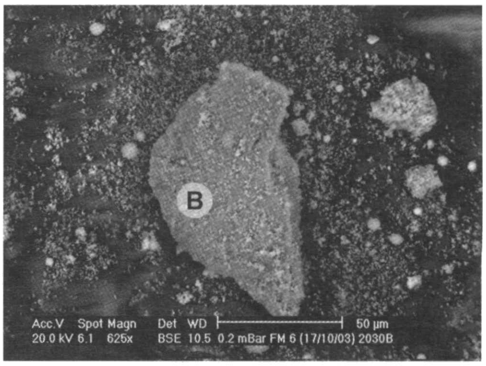

Figure 15. SEM photographs: waste RFT

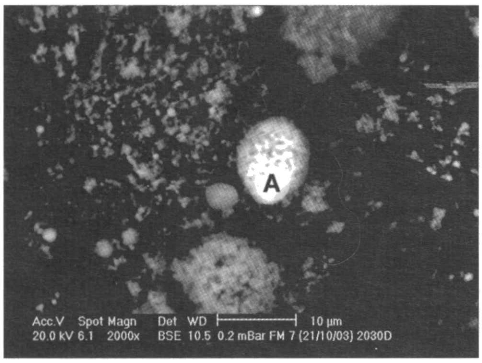

Figure 16. SEM photographs: waste RFO

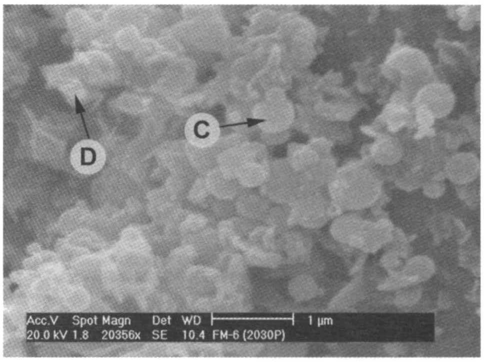

Figure 17. SEM photographs: waste RFT.

The photographs in Figures 14 to 17 show different types of particles present in the wastes held in the filters. Based on observation of the photographs and information in the literature[1-3][8], four types of particles have been distinguished, as follows:

- **Type A particles (coarse and rounded):** These are large-size particles (D>10 μm) with rounded edges and a glassy surface that can have a more or less important deposit of smaller crystalline and glassy particles. EDXA microanalysis of these particles indicates that as a whole, the spheres with surface deposits present the typical composition of a frit with relatively high quantities of zinc, calcium and potassium (Figure 14). The origin of these large spherical particles can be due to a crystalline particle acting as a condensation nucleus for volatiles, finally acquiring a rounded shape as a result of the presence of a liquid phase at the surface that gives rise to a glassy phase after cooling. Figure 16 clearly shows how this type of rounded particle has formed on a crystalline zirconium silicate particle.
- **Type B particles (coarse and irregular):** These are large-size particles (D>10 μm) with an irregular shape, whose composition suggests they are crystalline particles, concretely of quartz in Figure 15, with a diameter of the order of 50 μm.
- **Type C particles (fine and spherical):** These are small-size spheres (D<2 μm), whose chemical composition suggests they are made up of glassy particles rich in alkalis and chlorine, with a possible origin as condensed volatiles that give rise to microspheres (Figure 17).
- **Type D particles (fine and irregular):** These are small-size irregular coarse particles (D<2 μm), whose chemical composition suggests they are made up of fine crystalline particles like zinc oxide or quartz, which have been able to reach this stream on being swept along by gases (Figures 14 and 17).

Type A and type C particles are the particles that can pose most problems from the point of view of treatment system operation, in the circuit regions of greatest temperature (before the heat exchanger). These particles, which at ambient temperature display a spherical shape, have a semi-molten external surface at high temperatures, which can give rise to problems on sticking in ducts, heat exchanger valves, etc.[1]

5.3. CHARACTERISATION OF THE GASEOUS STREAMS

While the kilns were running according to the conditions detailed in Table 2, we characterised the gaseous streams from each kiln (before the filter) and the final treated stream. The particle concentration was calculated in actual emission conditions and related to two reference oxygen percentages, using Equation (1), to enable comparing the values obtained with the legal limits. The use of Equation (1) in oxycombustion systems like the one being studied may be debatable. However, as no equation was found in the BREF, legislation or standards surveyed, it was decided to employ this generally used equation.

$$C_{ref} = C_m \cdot \left(\frac{20.9 - [O_2]_{ref}}{20.9 - [O_2]_m} \right) \qquad (1)$$

where:

- C_{ref}: Particle concentration relative to the reference oxygen percentage (mg/Nm^3)
- C_m: Particle concentration (mg/Nm^3)
- $[O_2]ref$: Reference oxygen concentration (%)
- $[O_2]m$: Oxygen concentration measured in the gaseous stream (%)

The results obtained in the present study are detailed in Table 5.

Emission characteristics	Units	Before the filter		After the filter
		Kiln 6	Kiln 7	Stack
Composition				
O_2 content	% by volume	16.4	18.8	18.1
CO_2 content	% by volume	2.5	1.2	1.6
N_2 content	% by volume	81.1	80.1	80.3
H_2O content	% by volume	23.9	12.6	17.9
Flow rate and temperature				
Dry gases	Nm^3/kg frit	1.58	2.24	1.38 (*)
Wet gases	Nm^3/kg frit	1.96	2.52	1.63 (*)
Temperature	°C	173	143	120

(*) The output and input flow rates are not the same because there is a recirculating stream of cleaned air.
Table 5. Results of the emission characterisation.

To be noted in Table 5 are the low gas flow rates obtained per unit product. This was because the kilns were running with oxygen as an oxidising agent, and when the gas cleaning system was implemented, the amount of diluting air fed into the combustion gas stream was limited as much as possible. This highly reduced the emission flow rates and hence the dimensions of the heat exchangers, baghouse filters and running costs of the treatment facility. On the other hand, however, the pollutant concentration increased. This makes it necessary to achieve good cleaning efficiencies, if the emission limits expressed in terms of concentrations are to be met.

Table 6 has been drawn up on the basis of the foregoing results. This table compares the solid particulate emission values with the emission limits currently established at European Union level (BREF)[9] and in the legislations of the two European Regions where most frit and glaze manufacturers are located, namely the Valencia Region (Spain) and the Emilia Romagna Region (Italy)[1]. The document taken as a basis for the Valencia Region is the Agreement made in September 2003 between the Territory and Housing Authority (Conselleria de Territorio y Vivienda) of the Autonomous Government of Valencia and ANFFECC (Spanish National Association of Frit, Glaze and Ceramic Colour Manufacturers), which establishes the emission limits to be met by 31 December 2006.

As this table shows, the results obtained with the installed treatment system display an optimum efficiency in solid particulate removal, and the treated emissions of the studied facility meet the particulate emission limits set in the most stringent regulations that have recently come into force.

At the same time the table shows that reducing the emission flow rate brings the emission values expressed in terms of concentrations (mg/Nm^3) closer to the emission limit values, than when these are expressed as specific emission values (kg/Mg). In fact the BREF itself envisages this option for the glass industry[9], especially in those cases where oxygen is used as an oxidising agent, as in the studied system.

Legal reference	Solid particulate emission limits	Emission in the studied facility
BREF: frits	0.1 kg/Mg frit	0.006 kg/Mg frit
BREF: frits. Continuous kiln	30 mg/Nm^3 (8% O_2)	17 mg/Nm^3 (8% O_2)
ANFFECC – Autonomous Government of Valencia Agreement	40 mg/Nm^3 (15% O_2)	8 mg/Nm^3 (15% O_2)
Emilia Romagna (Italy) legislation. Frit kilns	30 mg/Nm^3	4 mg/Nm^3

Table 6. Comparison of measured emission values with specific legal limits for frit kilns

6. CONCLUSIONS

- A cleaning system has been designed, implemented and fine-tuned for gaseous emissions from ceramic frit melting kilns based on the use of baghouse filters. The system operates at a relatively low filtration temperature (of the order of 150°C), with a view to achieving a high removal rate of particulate pollutants in the gaseous stream.
- The results obtained in the gaseous emission characterisation tests indicate that high efficiencies are achieved in solid particulate removal. The system therefore enables conforming to the most stringent emission limits demanded in the European Union for this type of industrial activity.
- In systems like the one studied, which run on oxycombustion, low specific gas emission volumes are produced, which for the same pollutant emission values (kg/h) give rise to higher emission values when these are expressed in terms of concentration (mg/Nm^3). For this reason, to evaluate the efficiency of these facilities, it is recommendable to express the emission limit values as specific emission rates (kg/Mg), as envisaged in the BREF for the glass industry[9].
- The cleaning system allows in situ valorisation of the wastes obtained in the cleaning system, by introducing all these wastes as raw materials in the same frit manufacturing process, as a result of which the implemented gas treatment system generates no wastes.

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