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Household oven self-cleaning surfaces via catalytic thermal oxidation

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ABSTRACT

The present work is aimed at developing oven walls with self-cleaning properties, via catalytically enhanced thermal oxidation of soiling material within the standard range of household oven, temperatures, i.e. up to 300 ℃. Some saturated fatty acids, e.g. myristic (tetradecanoic), palmitic (hexadecanoic) and stearic (octadecanoic) acids, were selected as single soiling compounds to investigate their thermal degradation behaviour under different operating conditions.

 $MnCr_2O_4$, LaFeO₃ and CeO₂ were chosen as oxidative catalysts and synthesized by the "Solution Combustion Synthesis" (SCS) method. The catalytic activity towards fatty acids was tested in a temperature programmed combustion (TPC) apparatus. The Ozawa method was adopted to calculate the activation energy for the catalytic combustion of palmitic acid on, e.g. ceria showing a very low value of 50 kJ/mol compared to the 90 kJ/mol for the non-catalytic combustion. The prepared catalysts were then deposited on steel sheet samples coated with commercial enamel by *in situ* spray pyrolysis. The obtained catalytic layers were characterized by SEM–EDS analysis to assess their integrity and adhesion to enamel and the obtained phases. The self-cleaning performance of the coated sample activity was measured in a standard oven, by performing a cooking cycle simulation (250 ◦C for 60 min; soiling by olive oil and pork lard); the weight loss and aesthetic appearance were benchmarked versus a bare, non-catalytic enamel surface. The CeO2 catalyst showed the best adhesion properties as well as the best performances towards the combustion of the three selected fatty acids, whereas the $MnCr₂O₄$ catalyst provided the best performance towards both the pork lard and the olive oil catalytic combustion with fat removal per cycle of about 18% and 40%, respectively.

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1. Introduction

The domestic electrical appliances market is a well established business in which technological leaps related to primary functions have been absent from the scene for some time and competitiveness mostly concerns accessory added values. Nowadays, due to the globalization of the market, this situation is changing: those manufacturers who are willing to maintain the top positions in market share ranking are committed to searching distinctive innovations, possibly related to the main product mission [\[1,2\].](#page-5-0)

A primary area of improvement, which is directly linked to potential benefits for customers, is product usability. For household cooking appliances, oven cleaning represents a top priority improvement area. Oven soiling mainly consists of fatty acid splatters, thermally degraded by the prolonged exposure to oven wall temperatures (up to 300 \degree C), and closely attached to the walls themselves. Traditional cleaning processes are carried out under unfavourable ergonomic conditions, and for this reason, they are

often postponed. As a consequence, unpleasant odours during subsequent cooking cycles may arise along with an increasing thermal degradation of the soiling, which further increases the difficulty of their removal.

The household oven cleaning process is actually an issue for both customers and manufacturers. Traditional cleaning operations are today performed using suitable caustic detergents. An automated product has been implemented (usually known as the pyrolytic cleaning route) on high end products, in which organic remains are incinerated via high temperature ($>500\degree C$) cycles, lasting from 1 to 3 h, and are there easily removed as dust [\[3,4\];](#page-5-0) such a feature implies additional design difficulties and the use of special materials, which result in severe safety and cost issues. Furthermore, the energy consumption related to the high temperature process is very elevated.

The present work is aimed at investigating an improved automatic cleaning process, that is capable of satisfying both customer and manufacturer needs and significantly reduce manual operations, by providing a cheaper alternative to pyrolytic ovens but with a similar added value. This aim will be pursued by developing oven walls with self-cleaning properties, via catalytically enhanced thermal oxidation of soiling materials at temperatures within the

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available range of standard ovens in order to eliminate the soiling materials in real time during the cooking process.

2. Experimental

2.1. Catalyst preparation and characterization

Three different catalysts (CeO₂, LaFeO₃ and MnCr₂O₄) were prepared via a highly exothermic and self-sustaining reaction, the so-called "Solution Combustion Synthesis" (SCS) [\[5,6\]. T](#page-5-0)his technique is particularly suitable for the production of nanostructured catalytic materials. A concentrated aqueous solution of various precursors (metal nitrates as the metal oxide precursors and urea as the sacrificial fuel) was placed in an oven at 600° C for a few minutes in a crucible, in order to ignite the very fast synthesis reaction. The adoption of the SCS method entails the formation of pure oxide catalysts with rather high specific surface areas in the absence of any carrier.

All the catalysts were then ground in a ball mill and characterized by different analytical techniques. The achievement of the desired crystalline structure was investigated via X-ray Diffraction by means of a PW1710 Philips diffractometer equipped with a monochromator for the Cu-K $_{\alpha}$ radiation. A scanning electron microscope (Philips, Model 515) was used to analyze the microstructure of the crystal aggregates of the prepared catalysts, whose elemental composition was checked by EDS (energy dispersive X-ray spectroscopy). The BET specific surface areas of the prepared catalysts were measured by a Micromeritics ASAP 2010 analyzer.

2.2. Catalytic activity assessment

As previously mentioned, oven soiling is mainly generated by food residuals, in particular fatty substance splatters and condensed vapour. Fatty food substances can be categorised as vegetal fats, comprising mainly cooking oils (olive, seed, etc.), and animal fats, which include the fatty substances from animal meat (e.g. lard) and from creamery industries (e.g. butter). Due to the extreme variability in composition of natural fats and of their partial degradation after the cooking process, three synthetic fatty acids (myristic, palmitic and stearic), which are normally present in natural fats [\[7–9\], w](#page-5-0)ere selected as a benchmark for the purposes of the present work.

The selected fatty acids (Aldrich reagents, 99.8% purity) were characterized from the thermal degradation point of view, in order to understand their behaviour upon exposure to increasing temperatures in various conditions [\[10\].](#page-5-0) A thermal gravimetric analysis (TGA – Mettler-Toledo TGA-SDTA-851) was used to perform such a characterization according to the following experimental conditions: a sample of 50 mg was placed in the crucible and heated (heating rate of 10 \degree C/min up to 700 \degree C) with two different gas mixtures (air or 5 vol% of O_2 in N_2) at constant flow rate of 50 N ml/min.

The catalytic activity of the prepared catalysts was tested in the temperature programmed combustion (TPC) apparatus described in Ref. [\[11\]. T](#page-6-0)PC runs were subsequently performed for each catalyst/fatty acid combination, using the same procedure followed in the reference runs on bare acids. A TPC sample was prepared as a 1:9 (w/w) mixture of fatty acid and powdered catalyst, mixed in an agate mortar. This preparation leads to contact conditions between the catalyst and reagent that can be described as close; the interaction between the catalyst and reactant is in fact quite intensive, compared to what is actually achievable on an oven surface, but it enables a much higher degree of reproducibility, which is essential for activity screening studies.

The mixture (50 mg) was inserted, as a fixed bed, into a micro reactor between two layers of quartz-wool. The micro reactor was then placed in a PID-regulated oven and a K-type thermocouple was inserted into the bed. The tests were then carried out by heating the sample up to a target temperature (heating rate $5^{\circ}C/min$), where the combustion reaction was considered to be over; a mass flow controller delivered the controlled gas flow rate (air or the synthetic atmosphere with 5% vol. of oxygen) to the micro reactor.

The carbonaceous substance conversion was monitored, via a NDIR analyser (Hartman and Braun URAS 10E), by measuring the carbon dioxide and monoxide concentrations in the reactor exhaust gases. A computer recorded the fixed bed temperature and the $CO/CO₂$ outlet concentrations as a function of time. The $CO/CO₂$ outlet concentrations increased, starting from the carbonaceous substance ignition temperature, reached a maximum, and then decreased as a consequence of carbon consumption. For the catalyst activity experiments, the temperatures corresponding to the CO₂ peaks ($T'_{\rm PQ}$ and $T''_{\rm PQ}$) were taken as an index of the activity of the tested catalyst, while the CO release was qualitatively considered as an indicator of catalyst selectivity. The runs were all repeated three times and the average T_P value was assumed for each catalyst.

In order to fully appreciate the catalyst performance, blank TPC experiments, using air as the flowing gas, in the presence of only the inert $SiO₂$ particles (0.1 mm in size), were carried out for each fatty acid. After these experiments, T_{P0} and T_{P0}^* , defined as CO₂ and the CO maximum release peak temperature of the bare carbonaceous substance, respectively, could be identified.

A quantitative measurement of the catalyst effectiveness was the decrease in the activation energies for the fatty acid combustion reaction: Ozawa's method proved to be well suited to calculate the activation energy starting from experimental results obtained from differential thermal analysis (DTA). The differential thermal analysis runs were performed using PerkinElmer equipment in a temperature range from 50 to 720 \degree C with different heating rates (2, 5, 10, 15 \degree C/min) and under an air flow of 100 N ml/min. In this case, palmitic acid was used as a reference substance for the fatty acids.

In order to evaluate the activation energy for the non-catalyzed combustion reaction of palmitic acid, 10 mg of this compound was submitted to DTA runs with different heating rates. The catalyst effect on the combustion reaction was investigated by repeating the DTA runs, using 10 mg samples of a 9:1 by weight mixture of catalyst/palmitic acid (previously mixed for 15 min in a ball mill). Integration of the whole and portions of the DTA patterns allowed us to determine the temperatures corresponding to different percentages of carbonaceous material burned in each DTA analysis under different experimental conditions (heating rate, kind of carbonaceous sample, catalyst type). According to Ozawa's method [\[12,13\], t](#page-6-0)emperatures corresponding to the cumulative combustion of 25, 50 and 75 wt% of the total carbon-based material located in the sample holder (temperatures that for each kind of sample changed according to the heating rate) were derived, and used to calculate the activation energy.

2.3. Spray pyrolysis coating method

The spray pyrolysis process (SPP) is particularly suitable for the synthesis of *in situ* inorganic compounds over a flat, even surface, such as the internal surface of an oven [\[14\]. T](#page-6-0)he precursor solution was loaded in a compressed air spray gun, operating a 30° cone nozzle, with a pressure of approximately 5 bar. Enamelled steel samples (Ferro PercTM Enamel, from a stock drip pan) were heated to 500 °C (similarly to the previously described SCS experiments), and then sprayed with the precursor/combustible aqueous solution, with even shots lasting approximately 3 s, impacting the whole sample surface. The spraying time was chosen by observing the surface wetting degree, in order to avoid any excess precursor solution and subsequent droplet formation. The combustion reaction took place on the hot surface, and led to a catalyst layer that grew in intimate contact with the basic substrate, ensuring good adhesion. A subsequent heat treatment at 600 ◦C for 1 h was carried out to complete the combustion process and it allowed the full development of the catalyst crystalline structure. Even though the precursor chemistry and light-off temperature of the SPP process used were identical to those of the SCS technique, a significantly different microstructure of the catalyst was expected, due to different synthesis conditions and dynamics. In particular, the spray process potentially being uneven, the uniformity of the catalyst layers was investigated by observing different small areas, over several prepared samples. The same characterization procedure was adopted to analyze the microstructure and the elemental composition and to verify what the desired catalytic compound had been achieved over the SPP treated samples.

2.4. Catalytic coating activity assessment

The activity of the catalytic coated plates by SPP was evaluated and benchmarked using a food derived fat substance, namely pork lard. A solid substance was selected to avoid dripping during the experiment setup, which could have jeopardised the final results.

TPC experiments were performed using the following device and procedure: a 10 mm diameter quartz tube reactor, capable of hosting a strip of steel sheet coated with about 10 mg of pork lard evenly spread on the catalyzed surface with a spatula was adopted. A heating rate of 20 ◦C/min was set, to reproduce the one achievable in the stock oven used for the subsequent test campaign (described in the following paragraph), up to a target temperature of 300° C. A quite high air flow (100 ml/min) was set to simulate the forced air convection regime in the oven cavity during a ventilated cooking cycle; 60 min were necessary to complete the test.

Moreover, a real-scale test was designed as a final assessment of the presented study. A ventilated household oven (Hotpoint/Ariston Experience FZ 102 P1) was used as a real appliance for this purpose. A gravimetric experimental procedure was set up, in order to assess the catalytic performances of the coated plates. Square Enamelled steel sheet samples (approximately 100 mm per side) were coated with the three investigated catalysts using the SPP technique. Organic soiling was reproduced by using olive oil and pork lard, the latter coming from the same batch used for the TPC experiments. Reference samples, constituted by bare enamelled steel sheets soiled in the same way, were employed as baselines. The fatty substances were deposited on the catalyzed surface of the samples as evenly distributed drops of pork lard, previously melted at 60 ◦C. About 0.15 g of fatty substance was deposited on each catalyzed sheet sample using a syringe. The clean and soiled sample weights were measured (0.1 mg resolution) and recorded.

The soiled samples were placed on an oven grid, located at mid height in the oven cavity, and evenly arranged to ensure uniform heating (Fig. 1A). The cleaning cycle was based on three steps (Fig. 1B), followed by sample weighing: (1) a multilevel ventilated cycle with a heating rate of $20 °C$ /min up to $250 °C$, 1 h; (2) a barbecue cycle, set temperature 250 \degree C, 1 h; and (3) natural cooling, 30 min. The first step was intended to raise the temperature evenly in the samples by forced convection; the second step, with the grill heating element on, exploited direct IR irradiation of the soiled sample surfaces. The maximum allowed set temperature for the oven (250 \degree C) was used. The sample temperatures were checked at the end of the second step by an IR camera (FLIR ThermoVision A40), by quickly extracting the grid with the samples from the oven (Fig. 1A). A thermocouple was also fixed onto a dummy sample, centred between the others; the temperature was recorded over the whole cleaning cycle (Fig. 1B). After a proper cooling in the switched off oven, the samples were weighed again, at room temperature, and the residual, unburnt soiling material was evaluated

Fig. 1. SPP catalyst activity in a household oven procedure: (A) IR camera temperature measurement, (B) temperature profile on dummy sample.

through the difference in weight from the clean specimen. Finally, after each run, the samples were cleaned using a standard oven pyrolysis cycle (nominal temperature 500 ◦C), to check that the soil residuals were completely removed by gravimetric measurement.

The gravimetric measurement of the catalyst activity was evaluated by comparison with a reference baseline sample. The weight loss of a reference non-catalyzed sample can be related to fatty substance evaporation and partial degradation mechanisms that are obviously not connected to the catalyst activity. Such a weight loss was subtracted from the starting soil mass, in order to obtain the soil mass remaining after the cleaning cycle in the absence of catalyst. The activity of the catalytic coatings was hence evaluated in terms of percentage of the residual previously defined soiling material, detected through gravimetric measurement of the samples after each cleaning cycle.

3. Results and discussion

The TGA experiments of the selected acids highlighted a weight loss curve whose first derivative peak allowed the temperatures associated to the main weight loss to be identified. The decomposition temperatures and weight loss results obtained from the TGA experiments for different operative conditions are reported in [Table 1.](#page-3-0)

[Fig. 2](#page-3-0) shows the non-catalytic TPC run of the stearic acid as an example of the double step combustion process. The other two fatty acids showed very similar combustion trends. A weight loss of 80% occurred at $300-380$ °C, a temperature range in which the developed catalysts could work as oxygen pumps and promote catalytic oxidation [\[11,15–17\]. T](#page-6-0)he TPC plots were in good agreement with the TGA data, and, in particular, the multi-step combustion process was as evident as in the TGA experiments. Noticeably, $CO₂$ emission occurred in all cases involving a two-step pattern: the $CO₂$ peaks were named T'_{p_0} and T''_{p_0} . CO emission peak $T^*_{\text{p}_0}$ was essentially connected to the first CO₂ peak T'_{p0} : this suggested that the first step of combustion had much quicker kinetics, resulting in an

Fig. 2. Stearic acid TPC run: CO/CO₂ emission curves.

oxygen shortage and CO formation. All the three combustion processes were completed at temperatures of between 500 and 620 ◦C (Table 2).

It can be argued that during the first combustion stage (see Fig. 2) the direct oxidation of the organic compounds was accompanied by the formation of harder to oxidise intermediates which were then completely oxidised in the second combustion stage. This second stage in fact occurs at the typical temperatures at which carbonaceous compounds usually burn [\[11\].](#page-6-0)

As far as the catalyst characterization is concerned all the prepared catalysts were found to be well crystallized in the XRD analysis (not reported). All the main diffraction peaks listed in the reference JPCDS cards (LaFeO₃: PDF 75-0541; MnCr₂O₄: PDF 75-1614; $CeO₂$: PDF 65-2975) were observed. The FESEM analysis results (not reported) highlighted a very foamy structure for the three catalysts, a typical feature of catalysts synthesized by SCS. The crystal size of the considered catalysts ranged from between 60 and 100 nm, which is in substantial agreement with the specific surface areas measured (see Table 3).

The $CO₂$ and CO emission profiles detected in the TPC runs on the catalyst–fatty acid mixtures (see Fig. 3 for those related to the LaFe O_3 TPC experiments) show a severe change in the combustion reaction mode: the multi-step combustion process disappeared, resulting in a single-step process. Considering the mean values of $T'_{\rm PQ}$ and $T''_{\rm PQ}$ (referred to as $T_{\rm PQ}$) in Table 2 as a reference for the whole

BET values of SCS synthesized powders.

two-step reaction experiment (420 \degree C for myristic acid, 360 \degree C for palmitic acid, 425 °C for stearic acid) and the achieved $T_{\rm P}$ values of the single-step catalyzed reaction (quite similar for all three acids), the difference $\Delta T_{\rm P}$ = $T_{\rm P0}$ – $T_{\rm P}$ can be considered as a parameter that can quantify the positive effect of the catalysts. The calculated peak shifts $\Delta T_{\rm P}$ of each catalyst are reported in [Table 4.](#page-4-0)

 $CeO₂$ showed the best performance, followed by LaFe $O₃$ and $MnCr₂O₄$. With the latter there was a deterioration as far as the palmitic acid is concerned; however, the $MnCr₂O₄$ catalyst exhibited the best selectivity towards $CO₂$ (about 92%).

The calculated activation energies for the bare and catalyzed palmitic acid oxidation are reported in [Table 5. T](#page-4-0)he obtained combustion activation energy reduction was perfectly in agreement with the TPC test results. This is in line with the ideal behaviour

Fig. 3. LaFeO₃ activity on selected fatty acids - TPC runs; (A) CO₂ emission graphics; (B) CO emission graphics.

Table 4

Catalyst activity evaluation: CO₂ on fatty acids: $T_{\rm P}$ and $T_{\rm P}^*$ table.

Fig. 4. FESEM views of the SPP CeO₂ layer lined on enamelled steel; (A) $10K \times$; (B) $100K \times$.

Table 5

Activation energy of palmitic acid combustion in air in the presence of the prepared catalysts, calculated using by Ozawa's method.

of a heterogeneous catalytic process, where the reactant is also in the solid state: in this case mass transport plays a minor role, being driven exclusively by geometrical contact conditions, which were set close, as previously explained. Moreover, the gaseous reactant (i.e. oxygen) was provided in large excess, in order to fully permeate the fixed bed reactor.

As far as the complete enamel–catalyst–soiling system, is concerned, the SEM images reported in Fig. 4 show the $CeO₂$ coatings after the SPP deposition process.

A general characteristic of all three coatings was a certain degree of grazing; surface cracks, generated from surface round holes,

Fig. 5. XRD diffraction pattern of $CeO₂$, LaFe $O₃$ and MnCr₂O₄ catalytic layers deposited by SPP on enamelled steel (\hat{c} : CeO₂ main peaks from JPCDS ref. no. 65-2975; $\S:$ LaFeO₃ main peaks from JPCDS ref. no. 75-0541; $*$: MnCr₂O₄ main peaks from JPCDS ref. no. 75-1614).

appear most probably because of the evacuation of the gaseous species generated during precursor decomposition. This grazing appeared to be more evident (showing loose flakes) for $MnCr₂O₄$, which showed some tendency to loose catalyst powder. In all three cases, a clearly developed foamy microstructure was present, under the form of 50–200 nm grains (Fig. 4B).

The XRD results showed (Fig. 5) that, in all cases, the expected crystalline catalysts were synthesized on the sample enamel surface. However, LaFeO₃ and MnCr₂O₄ were characterized by a lower degree of crystallinity compared to $CeO₂$ as a likely consequence of the higher complexity of the crystal structure. The bare enamelled steel surface spectrum was also investigated and used as a reference.

Fig. 6. SPP catalyst activity on pork lard – TPC runs: $CO₂$ (A) and CO (B) emissions.

Fig. 7. SPP catalyst activity towards (A) pork lard and (B) olive oil combustion in a stock oven: weight loss of the soiling material (referring to bare enamel as a reference) due to the catalyst effect.

The catalytic activity of the coated enamelled steel towards the combustion of pork lard is reported in [Fig. 6. T](#page-4-0)he effect of the catalytic coatings is evident, compared to the non-catalyzed plate. The CO and $CO₂$ emission plots showed that, at the selected target temperature (300 \degree C), the non-catalyzed pork lard combustion was relatively slow and prolonged well beyond the set experiment time, and a noticeable amount of carbon monoxide was developed. The catalyst assisted processes showed a significantly more favourable performance, resulting in a peak-shaped emission curve of both CO and $CO₂$, within the imposed test time of 60 min (40 min for CO). The beneficial effect of the catalyst layer therefore appeared to be remarkable, even under operative conditions that were very different from those in the first set of TPC experiments performed on powders. It is also worth noticing that combustion light-off occurred, especially for $CeO₂$, at temperatures of around $200 °C$

Fig. 7 shows the percent abatement of both pork lard and olive oil during the catalyzed combustion cycles in the oven.

A certain scattering of results was detected, as reported by the error bars, attributed to the unavoidable limitations of test reproducibility. The tested catalysts, activated via the previously described thermal cleaning cycle, removed more than 15% in weight of pork lard (CeO₂ and MnCr₂O₄), and up to 40% of the residual soiling from the olive oil ($MnCr₂O₄$). All catalysts therefore showed clear activity towards carbonaceous soil decomposition under real use conditions, which paves the way for a completely passive cleaning system.

On such a basis, it is possible to foresee a significant reduction in the soiling accumulation rate as a consequence of the catalytic combustion effect that occurs during normal cooking cycles, even though, a periodic cleaning cycle is probably required in order to maintain the catalytic activity. This is obviously related to the specific cooking habits. For instance, if a soiling cooking process is followed by several ones with a limited soiling production, complete self-cleaning could be achieved.

The regeneration cycle would be characterized by much lower temperatures (about 300 \degree C) than those typical usually used for the pyrolytic cycle (about 500 ◦C). Such a lower temperature would be easily achievable in a standard oven without expensive modifications of the insulating materials and components. However, as carbon monoxide emission appears to be a constant issue in high temperature pyrolysis, the exhaust chimney honeycomb catalyst usually used in pyrolytic ovens would still be needed, possibly tailoring the light-on temperature to ensure an effective action over the entire CO emission profile.

Finally, apart from the nature of the catalyst, a key parameter governing conversion seems to be the geometrical contact condition between the catalyst and soiling material, which is favoured by a highly corrugated catalyst surface. This is probably the reason why the more corrugated $MnCr₂O₄$ spinel layer ensured the best performance under real operating conditions (Fig. 7). However, surface roughness may also lead to a scarce adhesion. This aspect requires an optimization trade-off.

4. Conclusions

The catalytic combustion of fatty acids using oxidation catalysts can be obtained in a shorter time with a drastic reduction of carbon monoxide production compared to non-catalytic combustion. The selected catalyst appreciably reduced the activation energy for the combustion of the organic substances selected to simulate a soiled oven.

The spray pyrolysis technique used to deposit the catalytic layer over the steel sheet previously recovered from alumina-silicates enamel, offered a good adhesion of the catalyst layer, especially when using the $CeO₂$ and LaFeO₃ catalysts. The spinel catalyst $MnCr₂O₄$ deposited over the steel sheet gave the best performance in terms of weight loss for olive oil (40%) and pork lard (18%) during a cooking simulating cycle at 250° C, a much lower temperature than the temperature used during a standard pyrolytic cleaning $(500 °C)$.

An optimised catalyst structure is currently being studied in an attempt to achieve both excellent adhesion properties and high catalyst surface roughness. However, taking into account the possible toxicity of chromium, ceria should be considered as the best compromise between activity and possible food employment.

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