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Synthesis of Fe/CaO active sorbent for CO_2 absorption and tars removal in biomass gasification

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ABSTRACT

CO_2 capture is a strategy to reduce gas emissions from hydrogen production process. Among different materials, CaO has been used for CO_2 absorption principally for its abundance and low toxicity giving economical advantage. In this work different Fe/CaO sorbents were prepared using three CaO precursors including CaO, $CaCO_3$ and $Ca(CH_3COO)_2{\cdot}H_2O$ and two iron salts, $Fe(CH_3COO)_2$ and $Fe(NO_3)_3{\cdot}9H_2O$. The effect of calcination temperature, CaO precursor, iron salt, and preparation conditions on the physico-chemical properties and CO_2 absorption capacity were discussed. To characterize the sorbents, various techniques such as XRD, BET and TPR and absorption capacity tests were performed.

The most appropriated calcination temperature to obtain CaO phase is 750 °C. The higher BET surface area, observed for sorbents prepared from calcium acetate, is correlated to the higher CO_2 absorption capacity. Iron (II) acetate promotes the formation of Fe_2O_3 phase and iron (III) nitrate leads to the formation of $Ca_2Fe_2O_5$. The mechanical mixture leads to the formation of Fe_2O_3 while sol–gel and impregnation methods promote the formation of $Ca_2Fe_2O_5$.

Finally the preparation from mechanical mixture of calcium and iron (II) acetates will be the best compromise between a high CO_2 absorption capacity and the presence of Fe^0 efficient for the tars reforming.

1. Introduction

In the last decades we have observed the harmful impact of CO_2 emissions to the environment and human lives. To reduce its emissions and avoid its effect, many different ways have been studied. Among those, we can mention separation techniques such as chemical absorption, physical absorption, membrane technology, chemical looping combustion and also sequestration methods, such as geological, mineral carbonation and ocean dump [1].

In industrial hydrogen production processes such as coal gasification, methane steam reforming, water gas shift (WGS) reaction and biomass gasification, the emissions of CO_2 can be reduced by CO_2 capture. In addition to the environmental impact, CO_2 removal from the reactor in biomass gasification process improves volatilisation and gasification steps and WGS and tar reforming reactions, shifting the balance towards the hydrogen production [2].

CaO sorbents are widely used in CO_2 absorption [2–4] due to their high stability in the carbonation and calcination (regeneration). However the carbonation and calcination reactions are not totally reversible and are related to conditions as temperature and pressure (Fig. 1 and Eq. (1)) [5,6].

$$P_{CO_2eq}\ (\text{atm}) = 4.137 \times 10^7 \times \exp\left(-\frac{20,474}{T\ (\text{K})}\right) \tag{1}$$

The CO_2–CaO carbonation reaction proceeds through two reaction steps. The first step is the rapid surface reaction and the second one is the slow diffusion of CO_2 through $CaCO_3$ layer formed during early stage of carbonation [7,8]. According to the micrograin model developed by Bouquet et al. [9], the first step of carbonation finishes when all the micrograins are filling and the carbonate layer reaches a critical thickness then in the second step CO_2 must diffuse at the level of the whole grain. These reactions can be affected by high temperature leading to sorbent sintering and also by the difference in porous volume between CaO and $CaCO_3$. As a result of these changes, CO_2 absorption capacity decreases.

In several works [10,11] different CaO based sorbents were prepared and tested with the aim to improve CO_2 absorption capacity. The incorporation of inert materials and the modification of CaO structure have been also studied [3].

CaO based sorbents used to CO_2 capture can be calcium based mineral such as limestones and dolomites or synthetic calcium based sorbents obtain from calcium hydroxides, calcium carbonates and calcium acetates. The properties of absorption and regeneration capacity of synthetic CaO sorbents can be influenced by precursors [4], preparation method, reacting atmosphere [3] and

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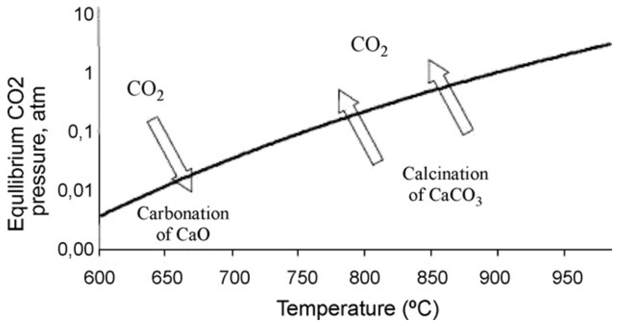

Fig. 1. Equilibrium curve for CO_2 capture.

calcination temperature. Lu et al. [4] identified the calcium acetate monohydrate as the best precursor of CaO sorbent because of its decomposition reactions. Authors attribute the formation of meso and macropores structures to the desorption of physically adsorbed gases and water, during the decomposition of calcium acetate to calcium carbonate above 400 °C and subsequent decomposition to CaO at temperature above 600 °C.

Furthermore in previous study, different CaO based sorbents have been synthesized on the basis of the integration of a metal, to obtain high conversion in tars reforming and high cyclic CO_2 absorption capacity [12]. The presence of iron could enhance the H_2 yield in the tars steam reforming from biomass gasification and in water gas shift reaction [13]. Simmell et al. [14] found that the presence of iron might improve the activity of carbonate rocks on decomposition of condensable tarry constituents of fuel gas. The use of iron as an active phase is attractive for industrial process due to its low cost, abundance and low toxicity. It is then important to invest in the knowledge of iron oxide states to determine its performance in steam reforming reactions and also its impact on CaO sorbents properties.

The aim of this work is to determine the optimal conditions such as precursor and preparation method to obtain Fe/CaO sorbent efficient for the CO_2 capture but also for tars reforming. They are required to have an absorption capacity adequate during repeated cycles of carbonation and calcinations and to easily form Fe^0 during reduction step. The identification of the phases obtained allows us to understand the effect of method and precursors on the performance of sorbents and metallic oxide role in CO_2 sorption process.

2. Material and methods

The Fe/CaO sorbents with 10 wt% of iron have been prepared by impregnation using an aqueous solution of precursor, mechanical mixture method performed in solid state and sol–gel synthesis based on thermal decomposition of propionates precursor [15]. Fe/CaO sorbents have been characterized by X-ray diffraction (XRD), Temperature Programmed Reduction (TPR) and BET method. CO_2 absorption capacities were then evaluated.

2.1. Sorbents preparation

Calcium precursors are CaO calcined at 900 °C (noted Caa), calcium carbonate ($CaCO_3$ noted Cab) and calcium acetate monohydrate ($Ca(C_2H_3O_2)_2 \cdot H_2O$ noted Cac). All these compounds were reagent-grade and purchased from Sigma Aldrich. In some cases it was necessary to calcine calcium precursors at 750 °C for better comparison and discussion; they are noted Caa_750, Cab_750 and Cac_750.

The iron salts used were iron (II) acetate anhydrous ($Fe(C_2H_3O_2)_2$ noted FeAc) and iron (III) nitrate ($(Fe(NO_3)_3) \cdot 9H_2O$ noted FeN) purchased from Alfa Aesar and Acros Organics, respectively.

2.1.1. Impregnation method (noted 1)

The iron acetate (FeAc) or iron nitrate (FeN) was solubilised in water; the calcined calcium oxide (Caa), calcium carbonate (Cab) or calcium acetate (Cac) was then added and stirred to obtain a suspension. After solvent evaporation at 70 °C, the sample is dried (120 °C, 12 h) and ground to fine powder. The material was then calcined at the desired temperature (600, 750 or 900 °C) for 4 h with a heating rate of 3 °C/min.

2.1.2. Mechanical mixture method (noted 2)

Mechanical mixture was carried out by crushing the iron salt (FeAc or FeN) and the sorbent precursor (Caa, Cab or Cac) in the adequate amounts until an homogeneous solid was obtained. The solid was calcined at 750 °C for 4 h with a heating rate of 3 °C/min. The particle size of the sorbent is in the range of 120–125 μm.

2.1.3. Pseudo sol–gel method [15] (noted 3)

The precursors used for this synthesis method were iron acetate (FeAc) and calcium acetate (Cac). The salts were separately dissolved in an excess of propionic acid. The solutions were mixed and heated under reflux. The solvent was evaporated to obtain a gel which was then calcined at 750 °C for 4 h with a heating rate of 3 °C/min.

2.2. Sorbents characterization

X-ray diffraction pattern of sorbents was recorded on an X-ray diffractometer Brucker AXS D8 Advanced using Cu Kα radiation to identify the crystalline phases. The diffraction spectra have been indexed by comparison with the Joint Committee on Powder Diffraction Standards (JCPDS) files.

BET surface area measurements were performed using nitrogen adsorption and desorption isotherms at −196 °C on a Micromeritics Tristar 3000 surface area analyzer. The CaO sorbents were previously calcined at 750 °C then degassed at 250 °C for at least 3 h.

The reducibility of the Fe/CaO sorbents under hydrogen was carried out on 100 mg of sorbent. First, decomposition of possible carbonates was performed from room temperature to 850 °C. The sorbent was cooled until room temperature and then the reductive gas mixture (10 vol% H_2 in Ar) passed through the reactor heated from room temperature to 900 °C with a slope of 15 °C/min then maintained at 900 °C for 30 min. A thermal conductivity detector (TCD) permitted the quantitative determination of hydrogen consumption.

2.3. Sorbents capacity

The absorption tests were carried out with 200 mg of sorbents by using a fixed bed reactor (6 mm of inner diameter). The CO_2 gas mixture (10 vol% CO_2 in argon) has a total flow of 50 ml/min and was controlled by mass flow controller. The absorption tests were performed in 4 steps. During the first step, the sorbent was heated until 850 °C under argon in order to desorb CO_2 and water molecules that come from atmosphere air, then the temperature was decreased until 650 °C (step 2). This temperature corresponds to the maximum temperature where CO_2 absorption still possible having in account Eq. (1) and Fig. 1 [5,6], and the minimum temperature for tars reforming [16]. At this temperature CO_2 absorption temperature was started (step 3): the CO_2 gas mixture was passed through the sorbent during 1 h. During desorption step (step 4), Ar (50 ml/min) replaced CO_2 gas mixture and temperature was increased from 650 °C until 850 °C with a heating rate of 5 °C/min. Desorption was followed by gas chromatography analysis of samples taken every 2 min until total disappearance of CO_2. The total desorption of CO_2 at 850 °C is then equivalent to the total CO_2 that could be absorbed in the sorption step (step 2).

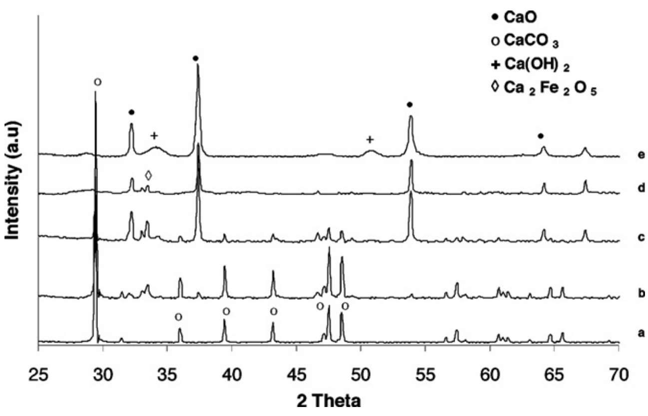

Fig. 2. Diffractograms of (a) $CaCO_3$ (Cab) and FeN_Cab1 calcined at (b) 600 °C, (c) 750 °C, (d) 900 °C, and (e) CaO (Caa).

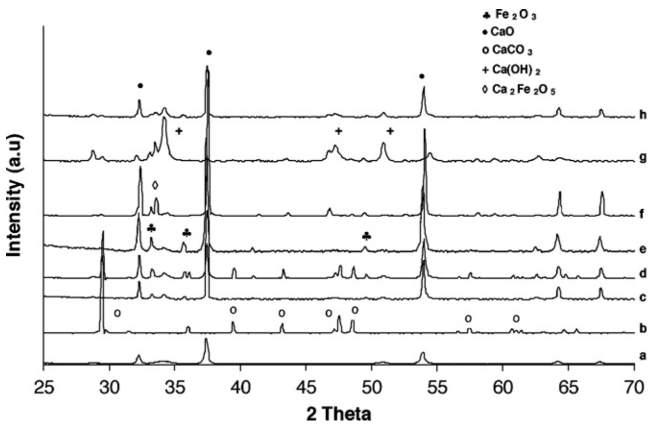

Fig. 3. (a) Calcium oxide (Caa), (b) calcium carbonate 750 (Cab), (c) FeAc_Caa2, (d) FeAc_Cab2, (e) FeAc_Cac2, (f) FeN_Caa2, (g) FeN_Cab2, and (h) FeN_Cac2.

3. Results and discussion

The sorbents studied were prepared taking into account different parameters (calcination temperature, calcium precursor, iron salt and preparation method) in order to obtain Fe/CaO solids with high performance in CO_2 absorption and tars steam reforming.

3.1. Effect of calcination temperature and iron presence

In order to determine the effect of calcination temperature on the phases composition of Fe/CaO, the sorbents prepared by impregnation of iron (III) nitrate on calcium oxide (FeN_Caa1) or calcium carbonate (FeN_Cab1) were calcined at different temperatures (600, 750 or 900 °C). The optimal calcination temperature was identified by XRD, BET and TPR analysis.

3.1.1. XRD

The FeN_Cab1 sorbents XRD results are presented (Fig. 2b–d) compared to those of calcium carbonate (Cab) and calcium oxide (Caa) precursors (Fig. 2a and e, respectively).

With an increase in calcination temperature, $CaCO_3$ is transformed into CaO. Although $Ca_2Fe_2O_5$ phase is also present, the contribution of this phase still minority and constant whatever the calcination temperature.

XRD patterns of FeN_Caa1 and FeN_Cab1 sorbents series differ only by the presence of $CaCO_3$ in the samples from calcium carbonate (Cab).

3.1.2. BET

It has been observed that CO_2 absorption can be affected by pores and surface areas [4]. The BET surface area of the iron sorbent calcined at 900 °C is lesser than that calcined at 750 °C (Table 1). With the sorbent calcined at 750 °C the CO_2 should diffuse more easily to reach the unreacted CaO in steam reforming conditions. Furthermore the presence of iron affects the surface area of CaO sorbent calcined at 750 °C (FeN_Caa1_750 compared to Caa_750, Table 1).

3.1.3. Temperature programmed reduction

To evaluate the effect of the calcination temperature on the interactions between iron and CaO phases, the TPR analyse was performed on the sorbents prepared by impregnation. Whatever the calcination temperature, the iron species are reduced at high temperature (800 °C). High calcination temperature leads then to the formation of strong interactions between iron (from iron (III) nitrate) and CaO. These iron species were previously identified as Fe^{3+} in the $Ca_2Fe_2O_5$ phase by XRD.

To conclude, the calcination temperature should be at least of 750 °C to obtain CaO. On the other side, higher calcination temperature leading to strong interactions between iron oxides and CaO and increasing the risk of metal sintering, a calcination temperature of 750 °C was adopted.

3.2. Effect of calcium precursor and iron salt

The different calcium precursors (Caa, Cab and Cac) and iron acetate (FeAc) or nitrate (FeN) were used to evaluate the effect of CaO precursor and iron salt. Mechanical mixture and impregnation methods were used and all sorbents were calcined at 750 °C.

3.2.1. XRD

XRD patterns of sorbent prepared by mechanical mixture show the presence of CaO (Fig. 3a) as principal phase whatever the CaO precursor (Fig. 3b–h). The calcination temperature is then well suited.

The difference in the structure of Fe/CaO sorbents is due to iron salt. When iron (II) acetate is used (Fig. 3c–e) hematite (Fe_2O_3) species are formed, and when iron (III) nitrate hydrate is used (Fig. 3f–h) the principal iron phase is brownmillerite ($Ca_2Fe_2O_5$). The $Ca(OH)_2$ and $CaCO_3$ species present were identified as impurities due to absorption of water and CO_2 from atmosphere.

Di Felice et al. [12] have observed that the presence of $Ca_2Fe_2O_5$ phase avoids iron availability for WGS reaction and tar reforming. Thus, we can expect that in the Fe/CaO sorbent prepared from mechanical mixture with iron acetate, iron will be more accessible (as an oxide and Fe^0) for reforming and WGS reactions and that calcium oxide will be available for CO_2 absorption. This result is a key to prepare a sorbent which reaches our objectives.

The effect of CaO precursor and iron salt was also studied by using impregnation method (data not shown). In this case CaO and $Ca_2Fe_2O_5$ phases are present in all sorbents even if iron (II) acetate salt is used. So it will be difficult to obtain Fe^0 in operating conditions. Here we have to point out the influence of iron salt and more particularly the iron oxidation degree and preparation method to prepare Fe/CaO sorbent with specific characteristics.

The particle size of both, sorbents from various precursors and iron sorbents were evaluated between 36 and 40 nm. We can conclude that the presence of iron or the preparation method did not affect significantly the particle size of the sorbents.

3.2.2. BET

The more important surface area was obtained when calcium acetate and iron acetate were used (Table 1). As we mentioned pre-

Table 1
Surface areas of CaO precursors calcined at 750 °C and effects of temperature, of calcium precursor and iron salt and of preparation method.

CaO precursors	BET (m^2/g)	Calcination temperature effect	BET (m^2/g)	Precursor and salt effect	BET (m^2/g)	Synthesis method effect	BET (m^2/g)
Caa_750	12.6	FeN_Caa1_600	2.1	FeAc_Cac2_750	8.8	FeAc_Cac1	7.2
Cab_750	12.7	FeN_Caa1_750	4.0	FeAc_Caa2_750	4.3	FeAc_Cac2	8.8
Cac_750	11.3	FeN_Caa1_900	0.1	FeN_Caa2_750	0.3	FeAc_Cac3	6.0

viously the acetate decomposition processes lead to a pores rich sorbent structure [3].

3.2.3. Temperature programmed reduction

The TPR profiles (Fig. 4) of sorbents prepared by mechanical mixture from the three precursors of calcium and two different iron salts (iron (III) nitrate or iron (II) acetate) show different reduction zones as a function of iron salt.

The sorbents prepared from the CaO or calcium carbonate precursors and from iron (III) nitrate (Fig. 4a and b) present a large reduction peak at high temperature (maximum between 817 and 836 °C). In these samples, XRD has permitted to identify the brownmillerite phase ($Ca_2Fe_2O_5$) as iron phase. Di Felice et al. [12] indicated that this phase, reducible at high temperature, leads to the formation of Fe^0. Therefore, TPR confirms the presence of this calcium–iron mixed oxide.

In contrast, in the sorbents prepared from calcium acetate and iron nitrate (Fig. 4c) or from the three CaO precursors and iron (II) acetate (Fig. 4d–f), TPR profiles of calcined sorbents indicate lower reduction temperatures (between 420 °C and 820 °C). Fe_2O_3 observed by XRD in these samples seems to be progressively reduced. The three first reduction zones can be attributed to the successive reductions of iron oxide (Fe^{3+} to $Fe^{2.5+}$, $Fe^{2.5+}$ to Fe^{2+}, Fe^{2+} to Fe^0) and, the fourth one, compared to the previous sorbents group, can be attributed to the reduction of $Ca_2Fe_2O_5$ to Fe^0.

XRD permitted to point out that the iron salts have an important effect on the iron species formed in the presence of CaO, and TPR indicated that the availability of iron in the sorbent is different according to the used precursor.

3.3. Effect of preparation method

According to the previous results, iron (II) acetate was chosen to determinate the effect of preparation method (impregnation, mechanical mixture and sol–gel). The calcium acetate is selected as calcium precursor because of its capacity to liberate water, acetone and CO_2 molecules at two different temperatures; therefore we expected to obtain the sorbents with the appropriate porosity to improve CO_2 sorption.

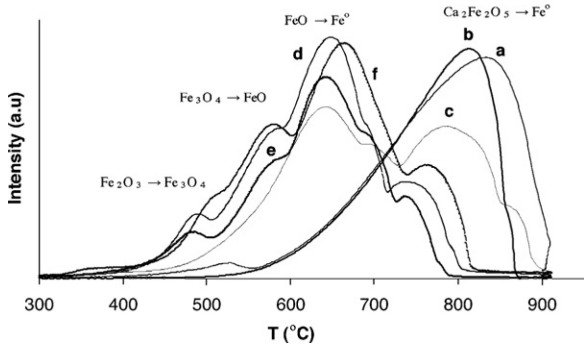

Fig. 4. TPR profiles – effect of CaO precursor and iron salt (a) FeN_Caa2, (b) FeN_Cab2, (c) FeN_Cac2, (d) FeAc_Caa2, (e) FeAc_Cab2, and (f) FeAc_Cac2.

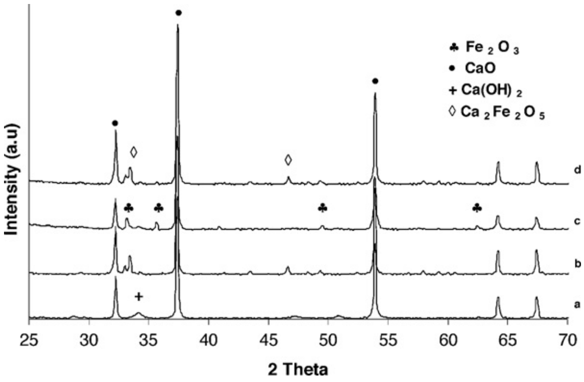

Fig. 5. X-ray diffractograms of CaO from acetate and Fe/CaO sorbents prepared by different methods and calcined at 750 °C. (a) Cac_750, (b) FeAc_Cac1, (c) FeAc_Cac2, and (d) FeAc_Cac3.

3.3.1. XRD

XRD patterns of Fe/CaO sorbents prepared by different methods are presented in Fig. 5. When sorbents are prepared by impregnation (Fig. 5b) or sol–gel method (Fig. 5d) the $Ca_2Fe_2O_5$ phase is the principal phase of iron. On the other hand sorbents prepared by mechanical mixture show Fe_2O_3 as principal iron phase (Fig. 5c). Then iron oxide (III) is only formed when calcium acetate precursor and iron (II) acetate salt are in solid state. It means that heating acetate precursors in solid state leading to the progressive release of water and acetone molecules, does not allow the formation of the undesirable $Ca_2Fe_2O_5$ phase.

3.3.2. BET

The specific surface (Table 1) of Fe/CaO sorbents is higher when they are prepared by the mechanical mixture (FeAc_Cac2). It seems to be the best sorbent for CO_2 capture.

3.3.3. Temperature programmed reduction

TPR profiles of precursors prepared by impregnation, mechanical mixture and sol–gel method are presented in Fig. 6.

The sorbent prepared by impregnation (Fig. 6a) and sol–gel (Fig. 6c) methods had showed by XRD that $Ca_2Fe_2O_5$ is present as

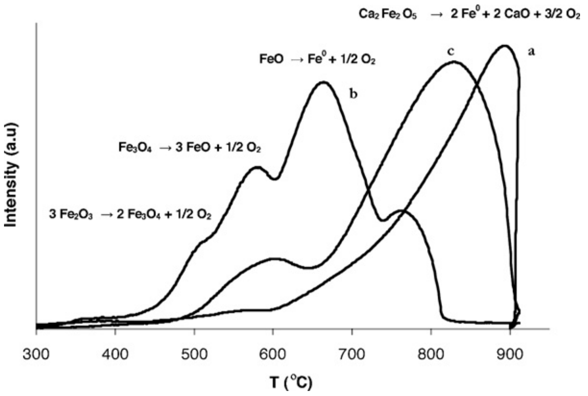

Fig. 6. TPR profiles – effect of preparation method (a) FeAc_Cac1 (b) FeAc_Cac2, and (c) FeAc_Cac3.

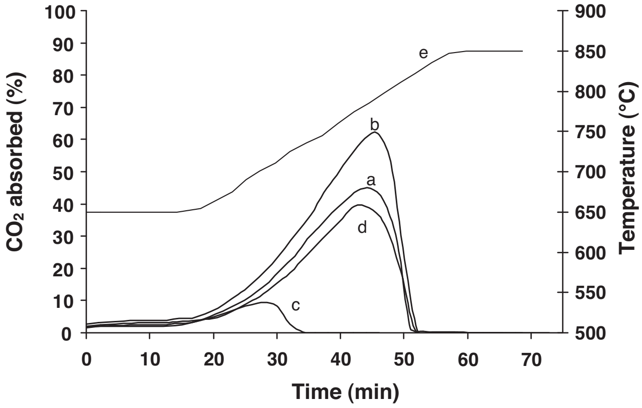

Fig. 7. CO_2 desorption with Fe/CaO sorbents prepared with different precursors. (a) Calcium oxide (Caa_750), (b) calcium acetate (Cac_750), (c) FeN_Caa2, (d) FeAc_Cac2, and (e) temperature ramp.

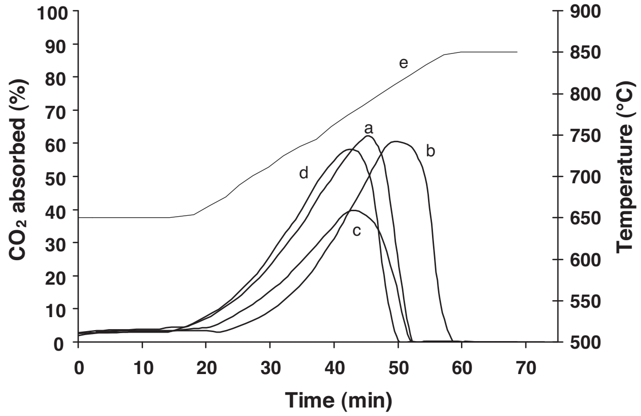

Fig. 8. CO_2 desorption with Fe/CaO sorbents prepared by different methods. (a) Calcium acetate (Cac_750), (b) FeAc_Cac1, (c) FeAc_Cac2, (d) FeAc_Cac3, and (e) temperature ramp.

principal iron phase; this result is confirmed by TPR. At high temperature the reduction of Fe^{3+} in brownmillerite phase to Fe^0 is complete in one step. A weak reduction peak is also observed near 600 °C for sol–gel method associated to the reduction of Fe_2O_3.

By mechanical mixture preparation (Fig. 6b), the typical Fe_2O_3 profile reduction is observed.

3.4. Sorbents capacity

In order to determine the efficiency of the most promising Fe/CaO sorbents, the CO_2 absorption capacity was evaluated and compared to the CO_2 absorption capacity of calcium precursors calcined at 750 °C (Caa_750 and Cac_750).

Desorption profiles are shown Fig. 7a and 7b for the calcium precursors. Cac_750 can absorb more CO_2 that Caa_750. Similar behaviour was reported by Lu et al. [4], for the sorbents prepared from calcium acetate and calcium oxide.

Fig. 7 permits also to evaluate iron presence effect on the absorption capacity by comparison of FeAc_Cac2 (Fig. 7d) to Cac_750 precursor (Fig. 7b). As mentioned before, XRD indicates the same range of particle size for all the sorbents then the absorption variations will not be due to this parameter. Iron decreases slightly the CO_2 absorption (25%). In contrast, the presence of iron, from nitrate, in FeN_Caa2 (Fig. 7c) sorbent decreases much more the CO_2 absorption capacity (77%) regarding to the Caa_750 precursor (Fig. 7a). In this case we can point out the importance of the selection of Ca precursor and iron salt, highlighting the use of iron acetate.

The use of different preparation methods, using acetate precursor (Fig. 8b–d), does not present a considerable effect on the sorption capacity of CO_2 (decrease of 4 and 8% of the CO_2 absorbed) compared to the support Cac_750 (Fig. 8a). The slight difference in the maximal temperature desorption could be explained by the little difference of the specific surface area. This result confirms the connection between physical properties (porosity) of sorbent and absorption capacities.

4. Conclusion

The conditions such as calcination temperature, CaO precursor and iron salt to obtain Fe/CaO sorbents with special characteristics for CO_2 absorption and tars reforming were evaluated. For example, when calcination temperature was 750 °C formation of CaO phase was enhanced. The sorbents prepared from calcium acetate have a higher BET surface and CO_2 absorption capacity than the sorbents prepared from CaO and $CaCO_3$.

Iron (II) acetate is the adequate salt to lead to the formation of Fe_2O_3 phase in Fe/CaO sorbents, thus iron can be more available for tars steam reforming. Mechanical mixture was identified as the best method to prepared Fe/CaO sorbents because it avoids the formation of $Ca_2Fe_2O_5$ phase.

The results obtained are a preliminary step to prepare the adequate Fe/CaO sorbents which will be used to study the decay of the CO_2 absorption capacity of sorbents in multiple carbonation and calcination cycles.

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