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Au/ZnO and Au/Fe_2O_3 catalysts for CO oxidation at ambient temperature: comments on the effect of synthesis conditions on the preparation of high activity catalysts prepared by coprecipitation

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The preparation of Au/ZnO and Au/Fe_2O_3 catalysts using two coprecipitation methods is investigated to determine the important factors that control the synthesis of high activity catalysts for the oxidation of carbon monoxide at ambient temperature. In particular, the factors involved in the preparation of catalysts that are active without the need for a calcination step are evaluated. The two preparation methods differ in the manner in which the pH is controlled during the precipitation, either constant pH throughout or variable pH in which the pH is raised from an initial low value to a defined end point. Non-calcined Au/ZnO catalysts prepared using both methods are very sensitive to pH and ageing time, and catalysts prepared at a maximum pH = 5 with a short ageing time (ca. 0–3 h) exhibit high activity. Catalysts prepared at higher pH give lower activity. However, all catalysts require a short operation period during which the oxidation activity increases. In contrast, the calcined catalysts are not particularly sensitive to the preparation conditions. Non-calcined Au/Fe_2O_3 catalysts exhibit high activity when prepared at pH ≥ 5. Calcined Au/Fe_2O_3 prepared using the controlled pH method retain high activity, whereas calcined catalysts prepared using the variable pH method are inactive. The study shows the immense sensitivity of the catalyst performance to the preparation methods. It is therefore not surprising that marked differences in the performance of supported Au catalysts for CO oxidation that are apparent in the extensive literature on this subject, particularly the effect of calcination, can be expected if the preparation parameters are not carefully controlled and reported.

KEY WORDS: gold catalysis; gold catalyst preparation; co-precipitation; iron oxide; zinc oxide.

1. Introduction

The observation that supported Au catalysts are highly effective for the oxidation of carbon monoxide at ambient temperature has prompted considerable research in this area [1–3]. High activity has been reported for a range of catalysts including Au/TiO_2 [4], Au/Mn_2O_3 [5], Au/ZnO [6] and Au/Fe_2O_3 [7]. Recently [8] the problems associated with reproducibility of the preparation of these materials has highlighted the need for standardised reference materials to provide benchmarks for activity. Catalysts have been prepared using an extensive range of methods [1–3], including impregnation, coprecipitation, deposition precipitation and vapour phase deposition. However, many preparation methods use different temperatures, pH and reaction times, and consequently materials with variable performance are prepared [8]. We have noted previously, that Au/ZnO catalysts prepared using coprecipitation were highly active when calcined, but showed poor activity when the calcination step was omitted [6]. In contrast, Au/Fe_2O_3 catalysts prepared similarly showed the reverse activity trends [7]. These observations have prompted us to explore the sensitivity of these catalysts to the method of preparation. To date there have been remarkably few comparative studies of catalyst preparation of Au catalysts for CO oxidation even though there has been intense research activity using these materials in recent years. Visco *et al.* [9] contrasted Au/Fe_2O_3 catalysts prepared using impregnation and coprecipitation and Chen *et al.* [10] have compared Au/Al_3O_3 catalysts prepared using deposition precipitation and chemical vapour deposition. Flytzani-Stephanopoulos and co-workers [11,12] have prepared Au/CeO_2 using deposition precipitation, coprecipitation and gelation methods and examined the catalysts for the water gas shift reaction. Schimpf *et al.* [13] and Haruta and co-workers [14] have prepared Au/TiO_2, Au/Al_2O_3 and Au/SiO_2 catalysts using a range of methods and evaluated the materials as hydrogenation catalysts. In these previous studies the distinct effects of catalyst preparation parameters have not been studied in detail. Very recently, Moreau and Bond have addressed this and have made an in-depth study of the Au/TiO_2 catalyst prepared by deposition preparation and they have shown that the preparation parameter can markedly affect the catalyst performance [15]. In this paper we

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report a preliminary study of the preparation of Au/ZnO and Au/Fe_2O_3 catalysts using two coprecipitation methods and demonstrate that the catalyst performance is highly dependent on the pH and the ageing time. Even short ageing times can have a marked effect, and consequently we use these results to comment on the problems associated with variable catalyst performance that has often been encountered in previous studies.

2. Experimental

2.1. Catalyst preparation

2.1.1. Preparation method A

Au/ZnO catalysts were prepared by a coprecipitation method using a variable pH method at constant temperature in which the pH of the precipitating solution was gradually raised. An aqueous solution $HAuCl_4.3H_2O$ (5 mL, 0.058 mol/L, Johnson Matthey) was added to an aqueous solution of $Zn(NO_3)_2{\cdot}6H_2O$ (100 mL, 0.1 mol/L, Aldrich) and was stirred at 80 °C. Aqueous sodium carbonate (0.25 mol/L, Aldrich) was added dropwise until a pH of 8.0 was obtained. The material was recovered by filtration and washed several times with cold and then hot water to ensure removal of Na^+ and Cl^-. After drying, initially at room temperature, and then in an oven at 90 °C for 16 h, samples were also calcined in static air at 400 °C for 3 h.

Au/Fe_2O_3 catalysts were prepared in a similar manner. An aqueous solution $HAuCl_4{\cdot}3H_2O$ (5 mL, 0.058 mol/L, Johnson Matthey) was added to an aqueous solution of $Fe(NO_3)_3{\cdot}9H_2O$ (50 mL, 0.1 mol/L, Aldrich) and was stirred at 80 °C. Aqueous sodium carbonate (0.25 mol/L, Aldrich) was added dropwise until a pH of 8.2 was obtained. The material was recovered by filtration and washed several times and dried/calcined in an identical manner to the Au/ZnO catalysts.

2.1.2. Preparation method B

Au/ZnO catalysts were prepared using a coprecipitation procedure at constant pH and constant temperature. An aqueous solution $HAuCl_4$ $3H_2O$ (5 mL, 0.058 mol/L, Johnson Matthey) was added to an aqueous solution of $Zn(NO_3)_2{\cdot}6H_2O$ (100 mL, 0.1 mol/L, Aldrich) and was stirred at 80 °C. Aqueous sodium carbonate (0.25 mol/L, Aldrich) was similarly preheated. The two solutions were then fed at a controlled feedrate to a thermostated precipitation vessel. The flowrates were adjusted so that the desired constant pH was maintained. Following the precipitation process the material was recovered by filtration and washed several times with cold and then hot water to ensure removal of the sodium and chloride ions. After drying initially at room temperature, the material was then dried in an oven at 90 °C for 16 h. Samples were also calcined in static air at 400 °C for 3 h.

Au/Fe_2O_3 catalysts were prepared in a similar manner using aqueous solution of $HAuCl_4{\cdot}3H_2O$ (5 mL, 0.058 mol/L, Johnson Matthey) and $Fe(NO_3)_3{\cdot}9H_2O$ (50 mL, 0.1 mol/L, Aldrich) maintained at 80 °C. Au concentrations were determined using atomic absorption spectroscopy.

2.2. Catalyst evaluation

The catalytic activity for CO oxidation was determined in a fixed bed quartz micro reactor, operated at atmospheric pressure. The feed consisted of CO/synthetic air (CO_2 free) with a molar ratio of 0.5/99.5. The combined flow rate was maintained at 20 mL min^{-1} and a constant catalyst loading of 50 mg was employed. The catalyst temperature was maintained at 25 °C by immersing the quartz bed in a thermostatically controlled water bath. Analysis of the reaction product was carried out on-line using gas chromatography. Standard catalysts supplied by the World Council [16] Au/Fe_2O_3 and Au/TiO_2 were also studied and these gave 95% and 100% CO conversion using our standard test conditions.

3. Results

3.1. Materials prepared by method A

3.1.1. Au/ZnO catalysts

A series of Au/ZnO catalysts were prepared using method A and evaluated as catalysts for the oxidation of CO at 25 °C. Initial experiments were carried out using a final pH = 8.0 to determine the optimum temperature for the preparation of active catalysts. The data presented in figure 1(a) are for the materials dried at 90 °C with no additional ageing step in the preparation, i.e., the material was filtered as soon as the pH = 8.0 had been reached. These data show that active catalysts can be prepared with temperatures in the range 60–80 °C, but at a higher precipitation temperature inactive materials are formed. In view of this we standardised subsequent preparations at a controlled temperature of 80 °C. The effect of the final pH was investigated at 80 °C and it is clear that pH has a marked effect on catalyst activity (figure 1(b)). Active catalysts can be prepared with a final pH in the range of 5–8, but a higher final pH led to inactive materials being synthesised. Analysis of the Au concentration showed that this decreased steadily as the pH was increased (pH = 5.0, [Au] = 6.5 wt%; pH = 6.0, [Au] = 4.5 wt%; pH = 7.0, [Au] = 2.8 wt%; pH = 8.0, [Au] = 2.6 wt%; pH = 9.0, [Au] = 2.0 wt%; pH = 10.0, [Au] = 1.9 wt%). Hence, part of the effect observed on catalyst activity is due to the decreased Au concentration of catalysts prepared at a higher final pH.

The effect of ageing was then examined at two pH values (pH 8, figure 1(c); pH 6 figure 1(d)). It is clear that ageing the material in this way prior to filtering had

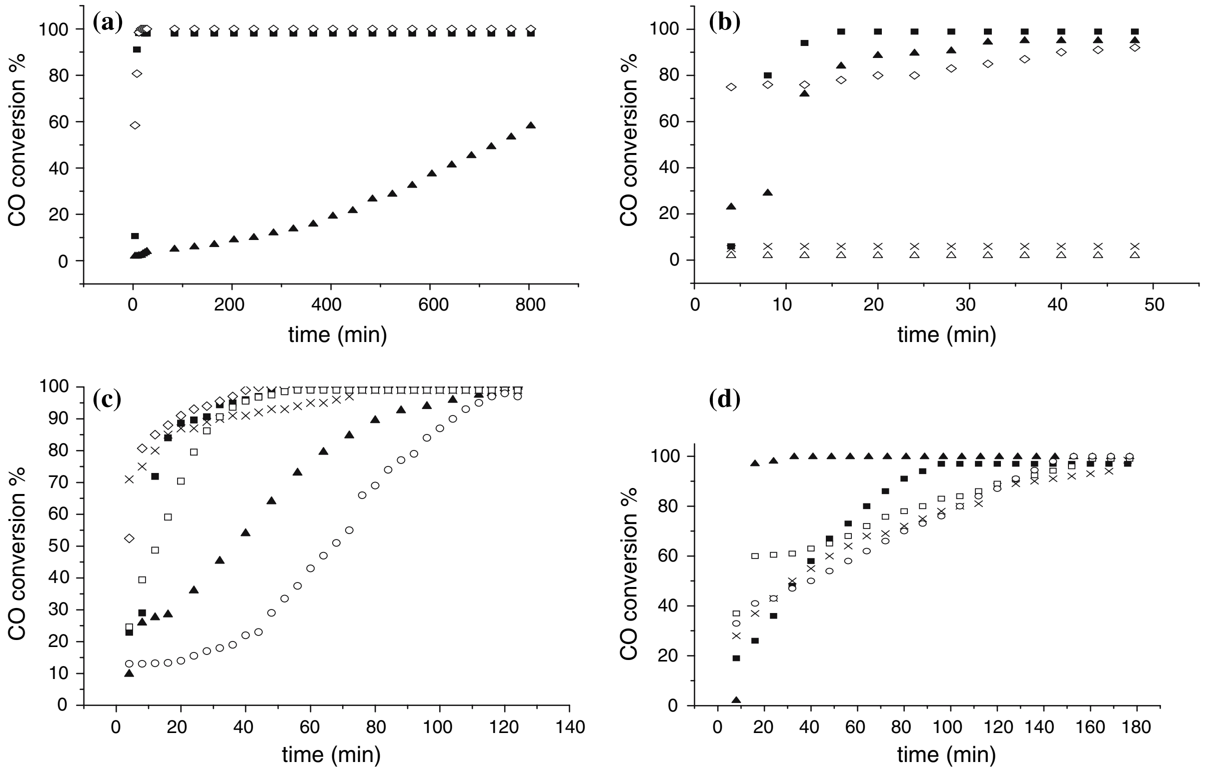

Figure 1. CO oxidation at ambient temperature using dried Au/ZnO catalysts prepared usingmethod A. (**a**) Effect of temperature of precipitation. Key: (■) 60 °C, (◇) 80 °C, (▲) 90 °C. (**b**) Effect of final pH for precipitation at 80 °C, Key: (■) pH 5, (◇) pH 7, (▲) pH 8, (×) pH 9, (△) pH 10. (**c**) Effect of ageing with a final pH 8.0 for precipitation at 80 °C, Key: (■) no ageing, (◇) 1 h, (▲) 3 h, (×) 5 h, (□) 8 h, (*o*)12 h. (**d**) Effect of ageing with a final pH 6.0 for precipitation at 80 °C. Key: (■) no ageing, (▲) 3 h, (×) 5 h, (□) 8 h, (*o*)12 h.

a marked effect on the initial activity of the dried material. Materials aged for aged for 1 h at pH 8 or 3 h at pH 6 show enhanced initial performance when compared for unaged materials. This shows a complex interplay between the variables of pH and ageing time. However, given sufficient reaction time all the catalysts eventually gave high conversion. It is clear that the catalyst structure is changing during the ageing process. Ageing of the catalyst for any time decreased the Au concentration, but the effect was independent of ageing time. At pH = 6.0 the Au content decreased from 4.5 wt% to ca. 3.5 wt%, whereas at pH = 8.0 the Au concentration decreased from 2.6 wt% to ca. 1 wt%. Since effective utilisation of Au is an important factor it is suggested that ageing of precipitates is not beneficial. However, calcination of the Au/ZnO catalysts prepared at different ageing times with final pH ≤ 8.0 all gave 100% CO conversion under our test conditions and this was stable for several hours and required no initial activation period. This finding is consistent with our previous study [6].

3.1.2. *Au/Fe_2O_3 catalysts*

Au/Fe_2O_3 material was also prepared using method A and the unaged material prepared at ph 8.2 and dried at 90 °C gave 100% CO conversion after 30 min time on stream, whereas the material calcined at 400 °C was relatively inactive (figure 2). However, the calcined sample was more active than Fe_2O_3 prepared in the absence of Au, indicating that the Au was still exhibiting a catalytic effect under these reaction conditions. These data are consistent with those observed in our previous studies with Au/Fe_2O_3 catalysts [7].

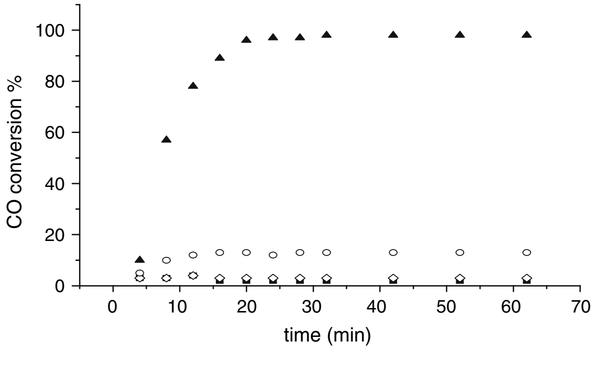

Figure 2. Activity of Au/Fe_2O_3 catalysts prepared using method A with a final pH 8.2 at 80 °C. Key:(■) Fe_2O_3 dried at 90 °C, (◇) Fe_2O_3 calcined at 400 °C, (▲) Au/Fe_2O_3 dried at 90°C, (*o*) Au/Fe_2O_3 calcined at 400°C.

3.2. Materials prepared using method B

Au/ZnO catalysts were prepared using method B in which the precipitation was carried out at constant pH (pH = 5.0–11.0 were investigated) and temperature (80 °C). The catalysts were evaluated as catalysts for the oxidation of CO at 25 °C, and the data presented in figure 3(a) are for the materials dried at 90 °C with no additional ageing step in the preparation, i.e., the material was filtered as soon as the required pH had been reached. The data show that the pH has a major effect on the activity of the dried materials. Active catalysts can be prepared when pH was maintained between 5 and 7, but at higher pH inactive materials were prepared. Calcination of the materials prepared at pH 5–7 at 400 °C gave catalysts that were active immediately, whereas the dried materials required a short activation time. Analysis of the Au concentration of the dried catalysts shows that as the final pH is raised so the concentration of Au significantly decreases (pH = 5.0, [Au] = 12 wt%; pH = 6.0, [Au] = 5 wt%; pH = 7.0, [Au] = 3 wt%; pH = 8.0, [Au] = 2 wt%; pH = 9.0, [Au] = 1 wt%; pH = 10.0, [Au] = 0.5 wt%), and hence the effect of pH is largely explainable in terms of its effect on the final Au concentration in the synthesised catalyst.

The effect of ageing was also investigated (figure 3(b)). In these experiments the precipitated material was left stirring in the reaction mixture at the final pH = 6.0 and at 80 °C for a specified time. The activity of the catalyst was found to be particularly sensitive to this parameter and catalysts with higher activity catalysts at short time on stream were observed when catalysts were aged in this way for a limited time (1 h) Ageing for longer times tended to give catalysts with poorer initial activity. However, for catalysts aged for ≥6 h the eventual activity (i.e., after ≥20–100 min time on line) did improve. Au analysis showed that using this method there was no significant effect of ageing on the Au concentration of the catalyst (i.e., at pH = 6.0 the Au concentration was ca. 5 wt% for all catalysts), which is in contrast to the effect of ageing on method A.

Au/Fe_2O_3 materials were also prepared using the constant pH preparation method without ageing of the

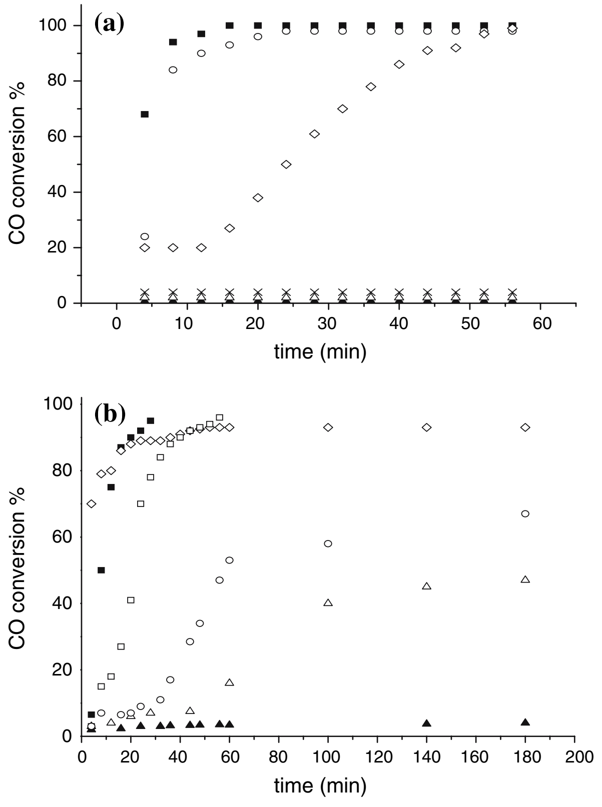

Figure 3. CO oxidation at ambient temperature using dried Au/ZnO catalysts prepared using method B at 80 °C. (**a**) Effect of pH. Key: (■) pH 5, (*o*) pH 6, (◇) pH 7, (×) pH 9, (△) pH 10, (●) pH 11. (**b**) Effect of ageing with pH = 6.0. Key: (■) no ageing, (◇) 1 h, (▲) 3 h, (△) 6 h, (□) 8 h, (*o*) 12 h.

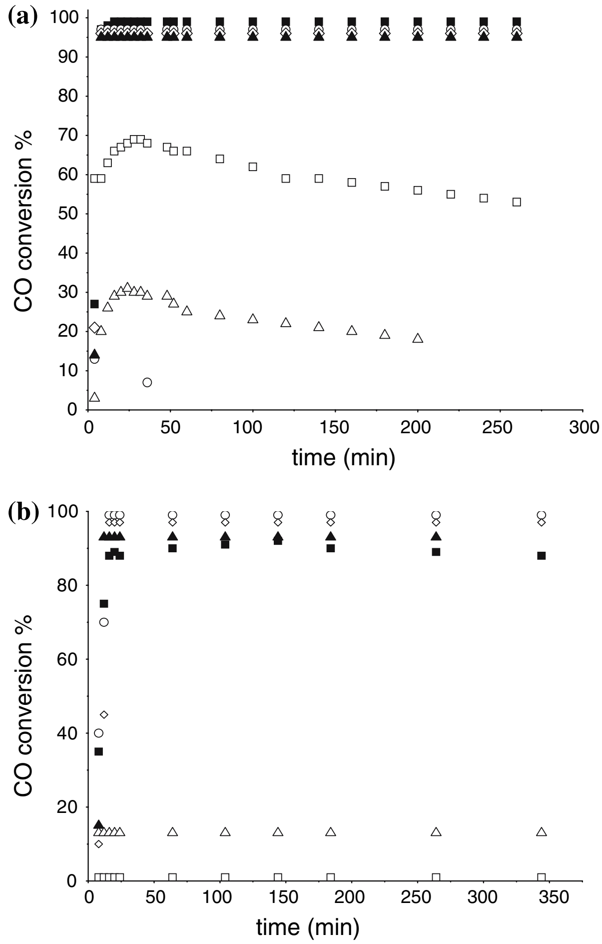

Figure 4. CO oxidation at ambient temperature using dried Au/Fe_2O_3 catalysts prepared using method B at 80 °C. (**a**) Effect of pH for dried catalysts Key: (□) pH 4, (■) pH 5, (*o*) pH 6, (◇) pH 7, (▲) pH 8, (△) pH 10. (**b**) Effect of pH for calcined catalysts. Key(□) pH 4, (■) pH 5, (*o*) pH 6, (◇) pH 7, (▲) pH 8, (△) pH 10.

precipitate. The dried materials showed that poorer activity materials were prepared at pH 4 and pH 10. (figure 4(a)). The materials calcined at 400 °C showed a similar activity pattern, except that the highest activity materials were prepared at pH ≥ 6 (figure 4(b)). The pH has an effect on the Au concentration of the final catalyst, at pH ≤ 8.0 the Au concentration of the synthesised material is ca. 9 wt%, but at higher pH only 0.5 wt% Au is precipitated. Hence, the poor activity of the catalyst prepared at pH $= 10$ is due to the low Au content. The poor activity of the catalyst prepared at pH $= 4.0$ is not, however, due to the Au concentration, and may be due to the microstructure of the support.

4. Discussion

The results presented in this study of the preparation of two materials Au/ZnO and Au/Fe_2O_3 show that high activity materials for the oxidation of CO at ambient temperature can be readily prepared for both calcined and non-calcined materials using two different coprecipitation procedures. These results are comparable with the results obtained from the standard catalysts supplied by the World Gold Council tested under the same reaction conditions. However, the most important observation is the potential sensitivity of these preparation methods to slight variations in the procedure when the materials are examined as catalysts for ambient temperature CO oxidation. For example, use of precipitation temperatures >80 °C led to inactive catalysts being prepared. Variation in the ageing of the precipitate prior to filtration, even for short periods of time, can significantly affect the observed activity, especially the initial activity. It has been noted that supported Au catalysts have given variable performance and that preparation methods can be very difficult to reproduce [8]. Our findings provide an answer to this putative problem, since in many published preparation methods a number of the preparation variables are not defined, especially the aspect of ageing of the precipitate. It is clear that small variations can lead to marked differences with the dried catalyst. Calcined catalysts are, however, not particularly affected by the reaction conditions, since the use of the elevated temperature leads to the formation of particulate Au with the same characteristics, and this is relatively insensitive to the starting structure.

Precipitation method A has been well studied previously and has been the main coprecipitation method used to prepare Au catalysts with ZnO and Fe_2O_3 as supports. Indeed, we have previously published a detailed transmission electron microscopy study of the materials made using this method for Au/ZnO and Au/Fe_2O_3 and correlated the data with the catalytic performance for CO oxidation [6, 7, 16, 17]. The present study has widened this early study and shows that active catalysts can be prepared for both dried and calcined materials as long as the precipitation conditions are closely controlled, in particular the pH, the temperature and the ageing time.

With precipitation method A the final pH is an important variable since the components present in the reaction mixture precipitate at different pH values [18]. For example, the onset of precipitation of Zn^{2+} is ca. pH 7, whereas the onset of precipitation of Fe^{3+} is ca. 3. Hence using precipitation procedure A the iron oxide/hydroxide/carbonates precipitates very early in the preparation procedure, and conversely the zinc oxide/hydroxide/carbonates precipitate relatively late in the procedure. This implies the preparation of Au/Fe_2O_3 materials using procedure A is similar to deposition precipitation since the support is precipitated prior to the active component. This is not the case for the Au/ZnO materials. However, it is quite clear that a calcination step ensures the formation of an active catalyst.

In addition to the effects of temperature and pH, catalysts prepared using both procedures A and B are very sensitive to ageing of the precipitate. This is a topic that has received very limited attention in the academic literature, and in particular has received very little attention for Au catalysts. However, it is recognised by commercial suppliers of oxide catalyst precursors that many basic carbonates and hydroxides that are initially precipitated during the coprecipitation process partially redissolve giving significant changes in morphology, and this is crucial for the eventual catalytic performance of the material. However, in the synthesis of most Au catalysts reported to date this potentially crucial preparation step has not been investigated or has gone unreported. Previously, we have shown that ageing plays an important part in the activity of Au/CuO/ZnO catalysts for ambient temperature CO oxidation [19]. Ageing of copper containing precipitates is known to be crucial and we have recently documented two cases in detail. First, we have shown the processes occurring in the transformation of CuO/ZnO precursors using detailed transmission electron microscopy, and demonstrated the effect on the activity for ambient temperature CO oxidation [20]. Also, we have shown that ageing can markedly affect the activity of $CuMnO_x$ hopcalite catalysts for CO oxidation [21,22].

In this study we have contrasted the catalytic performance of dried and calcined catalysts. For the dried materials in many cases the catalytic performance is observed to increase with time on stream, and sometimes this effect takes several hours. Although, eventually these materials usually gave 100% CO conversion. This gives further evidence as to why variable catalytic performance is often observed with supported Au catalysts, since their initial activity would be observed as poor, and, perhaps, it is not immediately apparent that the catalytic performance would improve with time on stream, since typically the reverse is observed. The calcined catalysts do not show this effect and they always give very high activities at the beginning of the experiment. Hence, this provides further evidence as to why most previous studies utilise calcination as a preparation step.

The origin of the activation of the dried catalysts may be due to two factors. First, the exposure of the catalyst to CO, a reducing gas may lead to the reduction of any cationic gold to metallic gold. Although it must be noted that a large excess of oxygen is present but gold is typically inert to oxidation by dioxygen. Second, the effect may be due to non-optimal levels of moisture being retained in the catalyst, since they are dried at 90 °C, a relatively low temperature. The presence of water is known to be crucial in the reaction of CO on Au nanocrystals. Very recently Haruta and co-workers [23] have shown that Au/TiO_2, Au/Al_2O_3 and Au/SiO_2 containing Au nanocrystals all increase in activity when low concentrations of water are co-fed. It is possible that during the initial phase of the catalytic reaction the optimal level of surface hydroxyl groups may be established for the dried materials.

The present study has shown that very active Au/ZnO and Au/Fe_2O_3 catalysts for CO oxidation at ambient temperature can be prepared without the requirement for a calcination step. The precipitation method is, however, very sensitive to the pH and the time that the precipitate remains in the presence of the precipitating solution. These findings are considered to throw light on the current problems that have been highlighted concerning the reproducibility of the preparation of high activity supported Au catalysts, since small variations in the way the precipitation is carried out can have significant affects on the both the initial and final catalytic performance. In this initial study we have concentrated on linking catalyst preparation parameters to the activity of the materials subsequently formed. We have not characterised the materials at this stage as this forms part of a subsequent study. However, the key observation is the sensitivity of the catalyst activity to subtle changes in the precipitation conditions.

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