



# Synthesis of Co<sup>3+</sup> Doped TiO<sub>2</sub> by Co-precipitation Route and Its Gas Sensing Properties

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Undoped and Co-doped TiO<sub>2</sub> nanoparticles were synthesized by a facile co-precipitation method and calcined at 700°C. The phase identification carried out by XRD measurements and Raman spectroscopy analysis of calcined powders reveals the formation of mainly anatase phase for undoped TiO<sub>2</sub>, and 0.5 mol.% Co-doped TiO<sub>2</sub> whereas rutile phase for 1 mol.% Co-doped TiO<sub>2</sub>. The sensors prepared with these powders deposited on interdigital (IDE) sensor platforms were tested toward NO<sub>2</sub> and H<sub>2</sub> sensing properties at 600°C. As the undoped and 0.5% Co-doped TiO<sub>2</sub> reveal n-type behavior, 1% Co-doped TiO<sub>2</sub> shows p-type semi-conductive behavior. One percentage Co-doped TiO<sub>2</sub> exhibits good sensing performance toward NO<sub>2</sub> while the undoped TiO<sub>2</sub> powder yields the best sensor performance toward H<sub>2</sub> at 600°C. This indicates that the crystal structure of TiO<sub>2</sub> sensing material must be adjusted depending on the nature of target gas. The results indicate that the main factor influencing high temperature gas sensor performance of nanoparticulate TiO<sub>2</sub> is either the alteration of its electronic structure or the type of polymorphs.

**Keywords:** co-precipitation, high-temperature sensor, nitrogen oxides, hydrogen, electronic structure, co-doped TiO<sub>2</sub>

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## HIGHLIGHTS

- Undoped and Co-doped TiO<sub>2</sub> were synthesized by co-precipitation route.
- Cobalt doping of TiO<sub>2</sub> promotes the anatase-rutile transformation.
- Undoped TiO<sub>2</sub> at 600°C, being n-type semiconductor shows the highest response to H<sub>2</sub>.
- 1% Co-doped TiO<sub>2</sub> with p-type behavior was more sensitive toward NO<sub>2</sub> at 600°C.

## INTRODUCTION

NO<sub>x</sub> gas sensors are gaining importance in automotive exhausts and combustion systems. Better performance resistive gas sensors can endure the harsh conditions and act effectively despite the strengthened international legislations. The corresponding gas sensors require durability at high working temperatures (exceeding >> 500°C). Semiconducting metal oxides are ideally used as gas sensing materials in resistive sensors due to their numerous benefits such as reasonably high sensitivity, easy fabrication processes and low cost (Dey, 2018). Most of transition metal oxides, such as SnO<sub>2</sub>, WO<sub>3</sub>, ZnO, NiO, or CuO used for this purpose display the optimum sensitivity at temperatures below 400°C (Wang et al., 2010). On the other hand, as one of the known

semiconducting oxides, TiO<sub>2</sub> is capable of operating as a gas sensing material at temperatures as high as 600°C (Esmailzadeh et al., 2012). The additional benefits of TiO<sub>2</sub> are non-toxicity, easy fabrication, and the good chemical stability (Chen and Mao, 2007). TiO<sub>2</sub> is a high resistive n-type semiconductor with rather poor conductivity to be adopted for sensing oxidative gases such as NO<sub>2</sub> (Huusko et al., 1993). Previous studies report that this disadvantage can be overcome by conversion of an n-type oxide to p-type semiconductor through addition of low valence dopant atoms to alter the electronic structure. Some previous studies in our team show how the Al and Cr-addition alters the electron conductivity of TiO<sub>2</sub> from n- to p-type leading to effective gas sensing materials. As far as Cr-doped TiO<sub>2</sub> is concerned, the optimum temperature for sensing properties has been limited with 500°C while the Al-doped TiO<sub>2</sub> detects NO<sub>2</sub> at 600°C but the sensitivity is low and the signal converts to p-type at 800°C (Saruhan et al., 2013; Gönüllü et al., 2015). Several other papers discuss the relation and reasons between the nature of the electronic structure and the sensing characteristics of the oxide materials (Wisitsoraat et al., 2009; Zhaohui et al., 2013; Gönüllü et al., 2015) and report that the enhancement of the sensing performance can directly be controlled by adjustment of the electronic structure to p-type at the semi-conducting oxide sensing layer.

In order to understand further if such an alteration can be achieved by Co-doping of TiO<sub>2</sub> and how this may affect the gas sensing behavior of TiO<sub>2</sub>, we have synthesized Co-doped TiO<sub>2</sub> nanoparticles by oxalate induced co-precipitation method and produced sensors by depositing the powders on interdigital electrodes. The resulting powders are characterized and the sensors are analyzed to identify their H<sub>2</sub> and NO<sub>2</sub> sensing capabilities. This paper reports, to the best of our knowledge for the first time, the effect of Co<sup>3+</sup> doping on the high temperature gas sensing behavior of TiO<sub>2</sub>.

## EXPERIMENTAL

Undoped and Co-doped TiO<sub>2</sub> nanoparticles were synthesized by employing the same processing route. Firstly, the starting precursor solutions were prepared. Cobalt acetate was dissolved in acetic acid as titanium iso-propoxide was diluted separately in absolute ethanol. The cobalt acetate and titanium iso-propoxide solutions were adjusted to obtain 0.5 and 1 mol.% of cobalt in TiO<sub>2</sub> and were labeled as 0.5 Co-doped TiO<sub>2</sub> and 1 Co-doped TiO<sub>2</sub>, respectively. These solutions were then mixed to each other and stirred for 5 min. Oxalic acid is utilized for the co-precipitation of a powder from this mixed solution. For that, oxalic acid was dissolved in absolute ethanol solution and poured progressively in to the mixed solution. The resulting mixture was stirred for 1 h to allow the complete precipitation. The so-obtained precipitate was filtered and dried in oven at 80°C yielding a white powder for the undoped and pink colored powder for the Co-doped samples. The as-prepared precursor powder was then calcined for 3 h at 700°C in a muffle furnace under static air.

The XRD diffractograms of the co-precipitated and calcined samples were collected with a D5000 Siemens Kristalloflex O-20 powder diffractometer having Bragg-Brentano geometry. The reflections from the JCPDS database were assigned to the experimental diffractograms by means of the program EVA from BRUKER AXS.

The Raman spectroscopy measurement were carried out at the Applied University of Bonn-Rhein-Sieg on Senterra Raman spectrometer from Bruker under 532 nm and 20 mW by focusing power laser excitation on samples through a 20 x objective. The morphology of the particles was determined by Scanning Electron Microscopy (SEM) analysis using a Zeiss Ultra 55 SEM.

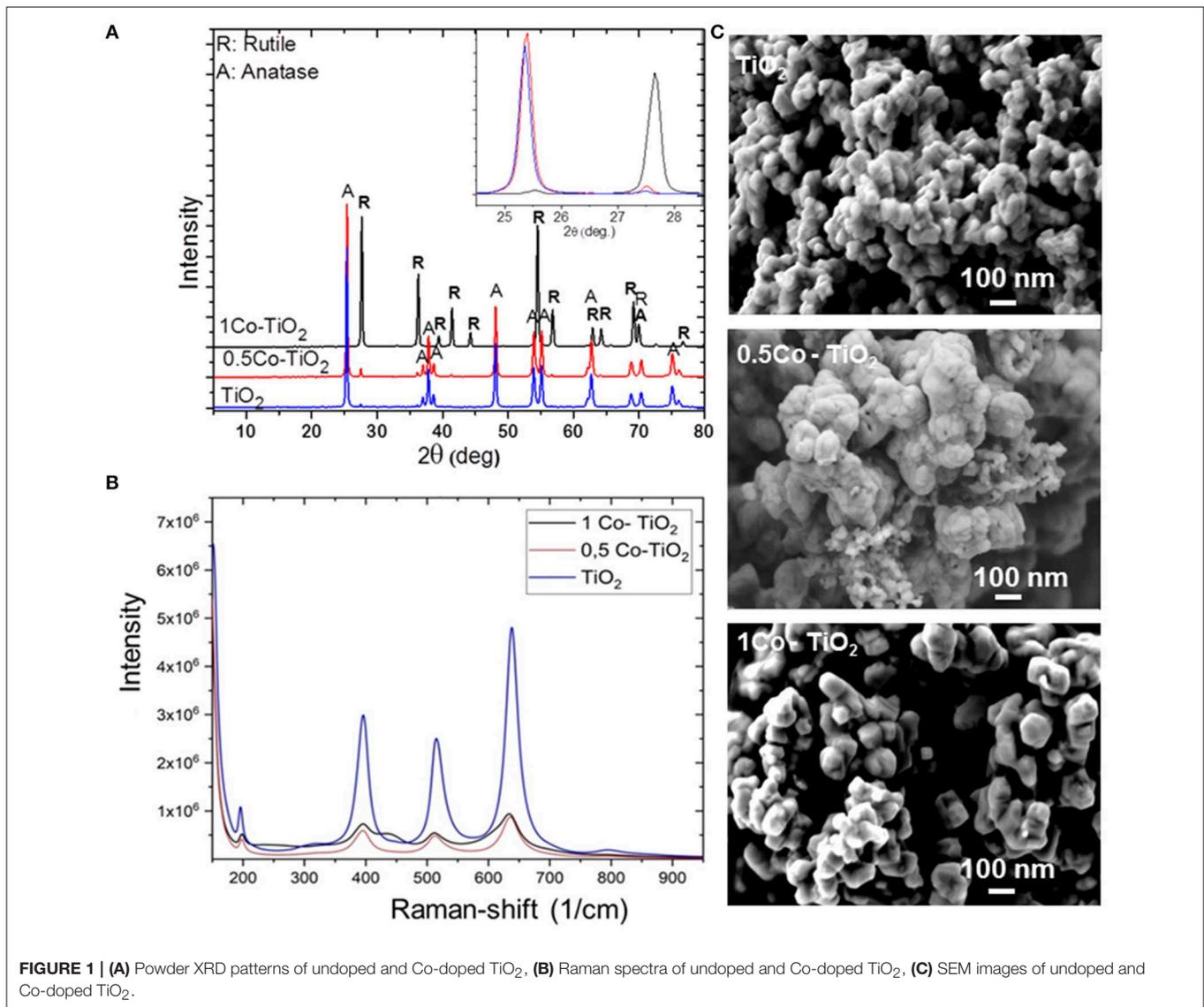
Specific surface area of the samples was determined using Micrometrics Tristar 3,000 equipment using N<sub>2</sub> adsorption at 77.150 K. Samples were degassed under vacuum at 120°C for 2 h prior to the surface area measurements. The specific surface area was calculated using the Brunauer-Emmet-Teller (BET) equation.

The as prepared TiO<sub>2</sub> and Co-doped TiO<sub>2</sub> powders were deposited as thick films using a simple drop-coating method on alumina substrates that were previously fitted with interdigitated electrodes. The sensor measurements were carried out in a specially constructed apparatus consisting of a tube furnace and a custom-built quartz glass reactor providing a thermocouple directed at the specimen. The electrical measurements were performed by using a Keithley 2635A Sourcemeter. The sensor response for n-type semiconductors is defined by  $(R_{\text{gas}}/R_{\text{air}} - 1) \times 100$  and  $(R_{\text{air}}/R_{\text{gas}} - 1) \times 100$  for oxidizing and reducing gases, respectively, while for p-type semiconductor,  $(R_{\text{gas}}/R_{\text{air}} - 1) \times 100$  and  $(R_{\text{air}}/R_{\text{gas}} - 1) \times 100$  for reducing and oxidizing gases, respectively.

## RESULTS AND DISCUSSIONS

The phase compositions of all synthesized powders were initially investigated by XRD as presented in **Figure 1A**. The undoped and the 0.5 Co-doped TiO<sub>2</sub> samples showed pure TiO<sub>2</sub> consisted of its two polymorphs; anatase (majority, JCPDS 21-1272) and rutile phases (minority, JCPDS 21-127). No other phase containing Co was observed. The amount of rutile phase was found to be slightly higher in 0.5 Co-doped TiO<sub>2</sub> than the undoped one. Also, the 1 Co-doped TiO<sub>2</sub> sample showed only single phase TiO<sub>2</sub> but this time the rutile polymorph was the major phase as anatase phase was in trace amount.

The X-ray results indicated that the cobalt dopant promotes the anatase-to-rutile phase conversion of TiO<sub>2</sub>. As presented on the inset of **Figure 1A**, a shift of the 2 Theta angles toward the higher values was observed indicating that the lattice parameters of TiO<sub>2</sub> decreases on Co-addition since according to the Bragg's law equation ( $2d\sin\theta = n\lambda$ ), the lattice parameter is inversely proportional to  $2\theta$ . Increasing  $2\theta$  angle does indeed mean that the lattice parameter decreases. In fact, the ionic radius of Co<sup>3+</sup> is smaller than that of Ti<sup>4+</sup> (Co<sup>3+</sup> = 0.685 Å, Ti<sup>4+</sup> = 0.745 Å). Hence, this leads to the contraction of the TiO<sub>2</sub> unit cell on substitution of Ti<sup>4+</sup> through Co<sup>3+</sup>. If otherwise, the substitution occurs through the incorporation of Co<sup>2+</sup>, an



opposite change in the lattice parameter would be expected (i.e., 2 Theta angles shift to lower values). Since Co<sup>2+</sup> has a larger ionic radius (Co<sup>2+</sup> = 0.79 Å) than that of Ti<sup>4+</sup>, an expansion of the TiO<sub>2</sub> unit cell shall occur yielding an increase of the lattice parameters. Chanda et al. has proven this assumption through their structural and magnetic studies on Co-doped TiO<sub>2</sub> (Chanda et al., 2018). Thus, it is plausible to consider in the present case that the substitution of Ti<sup>4+</sup> sites occurs by smaller ionic radius Co<sup>3+</sup> leading to a volume reduction of TiO<sub>2</sub>-lattice. Therefore, an overall volume contraction of ~8% is reasonable to obtain through Co<sup>3+</sup>-doping of TiO<sub>2</sub>. This promotes the conversion of anatase to rutile because the reconstructive anatase-to-rutile transformation involves a contraction of the c-axis (Hanaor and Sorrell, 2011).

**Figure 1B** displays the Raman spectra of the samples that were obtained between the wavenumbers of 200–900 cm<sup>-1</sup>. The Raman lines at 197, 390, 511, 637 cm<sup>-1</sup> can be assigned to E<sub>g</sub>, B<sub>1g</sub>,

A<sub>1g</sub>, or B<sub>1g</sub>, and E<sub>g</sub> modes of anatase phase, respectively, which confirm that the phase condition of our Co-doped TiO<sub>2</sub> samples belongs to the tetragonal form of anatase phase. Moreover, the spectra show that the peak intensities decrease drastically after doping. As XRD results showed that the main phase for 0.5 Co-doped TiO<sub>2</sub> is anatase, the decrease of intensity observed in the Raman spectra can be attributed to the increase of oxygen vacancies which occurred through the substitution of Ti<sup>4+</sup> by Co<sup>3+</sup> to balance the charge neutrality, relying on the difference between their ionic charges. These vacancies lead to the lattice distortion and results in a change of the diffraction intensity. Similar results were reported by Chanda et al. (2018). On the other hand, the XRD measurement revealed that the sample 1 Co-doped TiO<sub>2</sub> contains rutile as the major polymorph as anatase was only present in a very small quantity. This may explain the decrease observed at the Raman spectra of this composition. Moreover, the Raman spectra of 1 Co-doped TiO<sub>2</sub>

yielded a smaller shift toward lower wavelengths while new peaks ( $436\text{ cm}^{-1}$ ) appeared indicating the presence of rutile polymorph, as mentioned in literature (Niilisk et al., 2006).

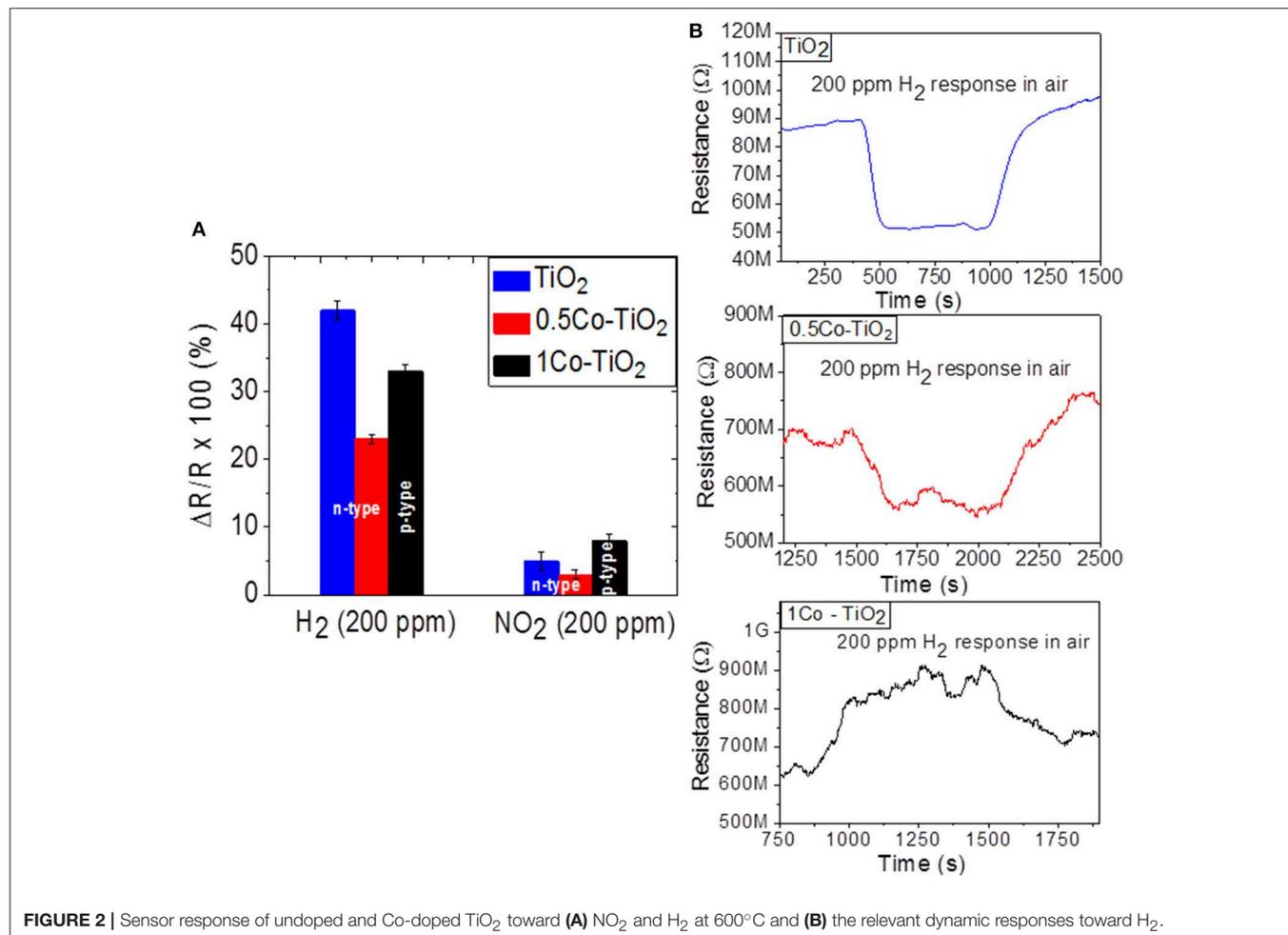
**Figure 1C** shows the morphology of the synthesized powders. The microscopic investigation revealed the formation of spherical nanoparticles with sizes around 70 nm that tend to agglomerate at the undoped powder samples. Doping with Co appears to influence the powder morphology. As the sample 0.5 Co-doped TiO<sub>2</sub> shows more agglomerated spherical particles, the sample 1 Co-doped TiO<sub>2</sub> presented larger and well-faceted rhombohedral crystallites with less agglomeration.

The BET-measured specific surface area of the synthesized samples were 8, 7, and 4 m<sup>2</sup>/g for undoped TiO<sub>2</sub>, 0.5 Co-doped TiO<sub>2</sub>, and 1 Co-doped TiO<sub>2</sub>, respectively. The surface area decreases with the increasing cobalt content, as the greatest decrease being with the 1 Co-doped TiO<sub>2</sub> that corresponds to the particle size increase as observed by SEM analysis (see **Figure 1C**).

The sensing properties of the undoped and doped TiO<sub>2</sub> were investigated toward 200 ppm NO<sub>2</sub> and 200 ppm H<sub>2</sub> at 600°C using synthetic air as carrier gas. This temperature

was chosen on the basis of our previous results obtained with undoped and Al- and Cr-doped TiO<sub>2</sub> relying on the fact that undoped TiO<sub>2</sub> is capable of NO<sub>2</sub>-sensing at above 400°C (Saruhan et al., 2013; Gönüllü et al., 2015). In fact, as the temperature increases, the sensor signal and also the response time decreases. Accordingly, our present results indicate that the equilibrium temperature for sensing is at 600°C. Additionally, it is known that the TiO<sub>2</sub> polymorphs will change at temperatures above 700°C. In order to avoid any temperature-induced complexity related to polymorph and electronic structures and to emphasize the effect of Co-dopant on the sensing properties, the operating temperature has been kept below 700°C.

As **Figure 2A** shows, the sensors yield higher response toward H<sub>2</sub> than NO<sub>2</sub>. The H<sub>2</sub> sensor responses are 42, 23, and 33% for undoped, 0.5 Co-doped TiO<sub>2</sub> and 1 Co-doped TiO<sub>2</sub>, respectively. The undoped sample shows the highest response toward H<sub>2</sub>. The presence of Co-dopant seems to decrease the H<sub>2</sub>-sensing performance of TiO<sub>2</sub> even though doping creates more oxygen vacancies in TiO<sub>2</sub>. This behavior can be attributed to the increase of rutile polymorph content on doping with cobalt, as previously reported, rutile is the less active (in term of functional properties)



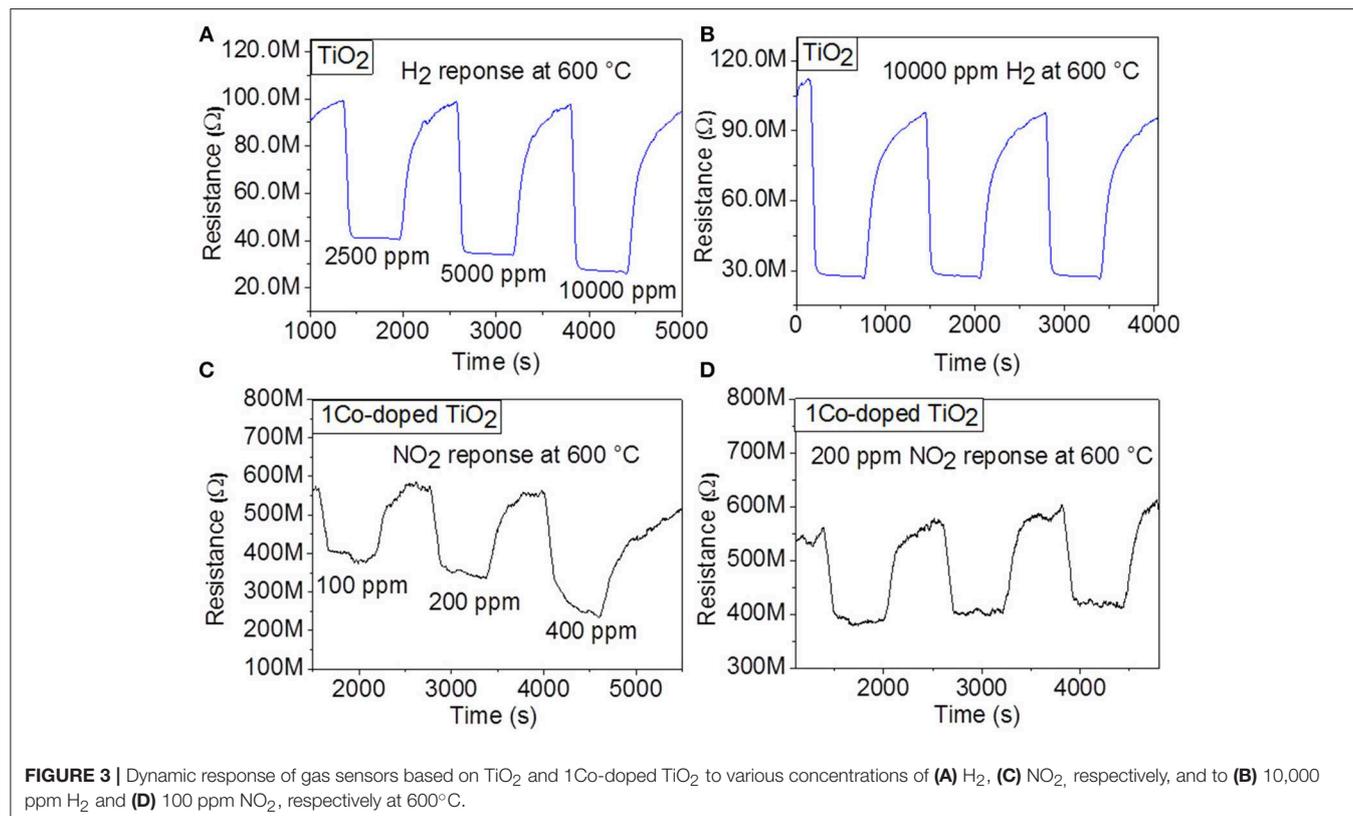
**FIGURE 2** | Sensor response of undoped and Co-doped TiO<sub>2</sub> toward (A) NO<sub>2</sub> and H<sub>2</sub> at 600°C and (B) the relevant dynamic responses toward H<sub>2</sub>.

polymorph of TiO<sub>2</sub> (Luttrell et al., 2014). On the other hand, the 1 Co-doped TiO<sub>2</sub> which contains predominantly rutile polymorph showed a higher response toward H<sub>2</sub> than 0.5 Co-doped TiO<sub>2</sub>. This discrepancy can be explained by alteration of the conductivity from n-type to p-type. In fact the dynamic response of the sensors toward H<sub>2</sub> given in **Figure 2B** reveals that undoped and 0.5 Co-doped TiO<sub>2</sub> exhibit n-type semi-conductivity (i.e., their electrical resistance decreases upon interaction with hydrogen) while the 1 Co-doped TiO<sub>2</sub> yields p-type conductivity (its electrical resistance increases when reducing gas is introduced).

The sensor responses measured toward the oxidizing gas NO<sub>2</sub> were 5, 3, and 8% for undoped, 0.5 Co-doped TiO<sub>2</sub> and 1 Co-doped TiO<sub>2</sub>, respectively. In the case of NO<sub>2</sub> sensing, the 1 Co-doped TiO<sub>2</sub> showed the highest response. This may be mainly due to the electronic alteration of TiO<sub>2</sub> from n to p-type semiconductor. Previous literature points out that this alteration can be utilized for the detection of oxidizing gas (Huusko et al., 1993). Our current results confirm that the dominant factor for the gas sensing property of the Co-doped TiO<sub>2</sub> depend on the existing polymorphs as well as the nature of target gas (oxidizing or reducing). In the case of reducing gases, the type of polymorphs has more influence on the gas sensitivity than the type of electronic structure, while an opposite trend can be observed for oxidizing gases.

Considering these preliminary results, sensing properties of the undoped TiO<sub>2</sub> toward H<sub>2</sub> and of 1 Co-doped TiO<sub>2</sub> toward

NO<sub>2</sub> are further investigated at 600°C. **Figures 3A,C** display the dynamic response of the sensor with undoped TiO<sub>2</sub> at 600°C to various H<sub>2</sub> concentrations (2,500, 5,000, and 10,000 ppm) and those with 1 Co-doped TiO<sub>2</sub> to various NO<sub>2</sub> concentrations (100, 200, and 400 ppm), respectively. As these response measurements exhibit, upon gas exposure, the sensor reaches to an equilibrium resistance value and settles back almost to the original baseline resistance value when the test gas is vented. This sequence has been reproduced for all the applied gas concentrations, demonstrating very good reproducibility and stability at the sensor behavior. For undoped TiO<sub>2</sub>, the calculated sensor signals were 58% for 2,500, 64% for 5,000, and 72% for 10,000 ppm of H<sub>2</sub>, while for 1 Co-doped TiO<sub>2</sub>, the sensing signal was 32% for 100, 40% for 200, and 55% for 400 ppm of NO<sub>2</sub>, respectively. These investigations indicate that the gas response increases with increasing concentration of NO<sub>2</sub> suggesting the capability of the sensor for quantitative analysis. In order to confirm the stability and reproducibility of the synthesized undoped TiO<sub>2</sub> and 1 Co-doped TiO<sub>2</sub> powders at the same H<sub>2</sub> and NO<sub>2</sub> concentrations, respectively, the sensor tests were repeated by introducing into the test chamber three times 10,000 ppm H<sub>2</sub> (for undoped TiO<sub>2</sub>) and 200 ppm NO<sub>2</sub> (for 1 Co-doped TiO<sub>2</sub>) under the same test conditions. The dynamic curves of these investigations are shown in **Figures 3B,D**. The average value of the gas response is 70% for H<sub>2</sub> and 38% for NO<sub>2</sub> for undoped TiO<sub>2</sub> and 1 Co-doped TiO<sub>2</sub> respectively, with a negligible deviation. Even though the baseline resistance changes slightly with time, the stability of the sensors is maintained well.



## CONCLUSION

This paper reports the successful synthesis of undoped and Co-doped TiO<sub>2</sub> nanoparticles by a facile co-precipitation route through the use of oxalic acid. According to the X-ray diffraction and Raman spectroscopy analysis, the substitution of Ti<sup>4+</sup> by Co<sup>3+</sup> creates oxygen vacancies and promotes the anatase-to-rutile transformation. The sample 1 Co-doped TiO<sub>2</sub> which reveals p-type conductive behavior yields an enhanced NO<sub>2</sub> response at 600°C under air as carrier gas. However, the undoped TiO<sub>2</sub> shows n-type semiconductor behavior with the highest sensor response toward H<sub>2</sub> at 600°C. The enhancement of high temperature NO<sub>2</sub> sensing performance is related to the alteration in electronic structure as well as the formation of rutile polymorph in the 1 Co-doped TiO<sub>2</sub> sample. It has been a remarkable effect to obtain two different behaviors by only addition of very small quantities of Co-dopant to TiO<sub>2</sub> under the same processing conditions. If the Co-dopant content exceeds 1%, CoTiO<sub>3</sub> starts to form as a secondary phase introducing a new parameter to take into consideration. For the perspective of NO<sub>2</sub> sensing

optimization, Co-dopant contents between 0.5 and 1 mol.% may also be considered.

## DATA AVAILABILITY STATEMENT

All datasets generated for this study are included in the manuscript/supplementary files.

## AUTHOR CONTRIBUTIONS

RL has synthesized and characterized the material for sensor application. BS have initiated the idea of applying the Co-dopant to TiO<sub>2</sub> and gave feedback in evaluating the sensing mechanism with this material.

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**Conflict of Interest:** The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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