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Fabrication of polypyrrole gas sensor for detection of NH_3 using an oxidizing agent and pyrrole combinations: Studies and characterizations

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ABSTRACT

The organic polymer known as Polypyrrole (Ppy) is synthesized when pyrrole monomers are polymerized. Excellent thermal stability, superior electrical conductivity, and environmental stability are all characteristics of Polypyrrole. Chemical oxidative polymerization was used to synthesize Ppy using Ferric chloride ($FeCl_3$) as an oxidizing agent and surfactant CTAB in aqueous solution. Oxidant ($FeCl_3$) to pyrrole varied in different molar ratios (2, 3, 4 and 5). It was found that increasing this ratio up to 4 increases PPy's conductivity. XRD, FTIR, and SEM were used to characterize Ppy. The conductive nature of Ppy was studied by I–V characteristics. The best conductive polymer is studied for the NH_3 gas response.

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1. Introduction

Without a certain class of materials known as polymers, life would appear to be quite challenging. Daily items like rubber, plastic, resins, adhesives [1], and adhesive tapes [2], are part of the ecosystem. The phrases poly and mers are Greek words that mean many and parts or units of great molecular mass. Each molecule in a polymer is composed of countless discrete structural components, that are strongly bonded. A polymer is generally called a big molecule which is highly weighted [3–5]. The process of joining monomers to create a polymer is referred to as polymerization. The observation can be seen in DNA, and RNA from the beginning of life [6–8], Additionally, carbohydrates and proteins play important functions in plant and animal life. Man has used naturally occurring polymers from the dawn of time to make tools, clothing, decorations, shelter, tools, weapons, writing materials, and other necessities. However, it is generally agreed that in the nineteenth century when significant discoveries were made regarding the modification of some natural polymers to get the kick start of the modern polymer industry [9].

The use of Polypyrrole (PPy) for gas sensing has been a topic of significant research in recent years, due to its high conductivity, stability, and potential for use in a variety of applications. The use of ferric chloride (FeCl3) as an oxidizing agent in the synthesis of PPy has been shown to further improve its electrical conductivity, and stability, making it a promising material for use in gas sensing applications.

The main novelty of using $FeCl_3$-doped PPy for the detection of ammonia (NH3) lies in its ability to respond rapidly, and selectively to the presence of NH_3 in the air, with changes in conductivity that are proportional to the NH_3 concentration. This represents a significant scientific contribution, as it provides a new and improved method for detecting NH_3, which is a common and toxic gas that can be found in many industrial settings, as well as in agricultural, and waste management systems.

In addition, the use of FeCl3, and pyrrole combinations in the synthesis of PPy has been shown to further improve the performance of the sensor. The $FeCl_3$ acts as an oxidizing agent, which enhances the conductivity of the PPy, while the pyrrole provides the necessary chemical reactivity for gas sensing. This combination of materials results in a sensor that is highly sensitive, selective, and stable, making it a promising alternative to traditional gas sensors based on metal oxide semiconductors.

In summary, the main novelty, and scientific contribution of using $FeCl_3$-doped PPy for the detection of NH_3 lies in its ability to provide a fast, accurate, and reliable method for detecting this toxic gas, with improved sensitivity, selectivity, and stability compared to other conductive polymer-based sensors. This study represents a significant step forward in the development of cost-effective, highly sensitive, and selective gas sensors based on conductive polymers.

Oxidation's conditions and reagents utilized have an impact on the material's conductivity. PPy's chemical composition is showing in Fig. 1.

In addition, Polypyrrole (PPy) is a conductive polymer that has been widely used in various sensor applications due to its high electrical conductivity, good stability, and biocompatibility [10–13]. To further enhance the performance of PPy-based sensors, composite materials are often synthesized by combining PPy with other materials, such as nanoparticles, carbon nanotubes, or graphene [14–16]. The synthesis of PPy composites can be achieved by different methods, including in situ polymerization, electrochemical deposition, and chemical oxidation polymerization [17–20]. Electrochemical deposition (ECD) is a commonly used method for the synthesis of Polypyrrole (PPy) composites [21–23]. In this method, PPy is deposited onto the surface of a filler material, such as nanoparticles, carbon nanotubes, or graphene, in an electrochemical cell. The deposition process involves the oxidation of pyrrole monomers at the anode and the reduction of pyrrole monomers at the cathode, resulting in the formation of PPy on the cathode [24–27]. In the electrochemical deposition of PPy composites, the filler material is first introduced into the electrochemical cell, and then a potential is applied between the electrodes to initiate the deposition of PPy [28,29]. The properties of the PPy composite can be controlled by adjusting the composition of the pyrrole monomer solution, the applied potential, and the duration of the deposition process. ECD offers several advantages for the synthesis of PPy composites, including simple and controlled fabrication, low cost, and good compatibility with various filler materials [30]. By electrochemically depositing PPy onto the surface of filler materials, PPy composites can be prepared with improved electrical conductivity, stability, and tunable properties compared to pure PPy. In summary, electrochemical deposition is a versatile and convenient method for the synthesis of PPy composites, and it offers opportunities for the development of new materials with improved properties for various applications, such as gas sensing, bio sensing, and energy storage. In situ polymerization involves the simultaneous polymerization of pyrrole and the incorporation of the filler material, while electrochemical deposition involves the growth of PPy on the surface of the filler material [31,32]. Chemical oxidation polymerization involves the oxidation of pyrrole in the presence of a filler material. The properties of PPy composites can be tailored by controlling the

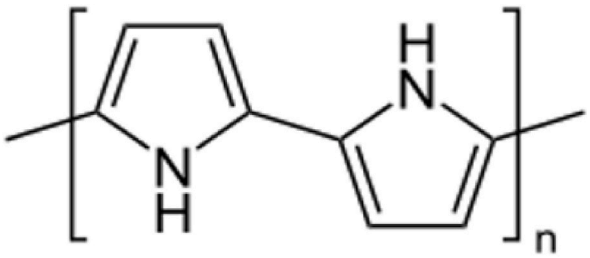

Fig. 1. Chemical Formula for Polypyrrole $(C4H2NH)_n$.

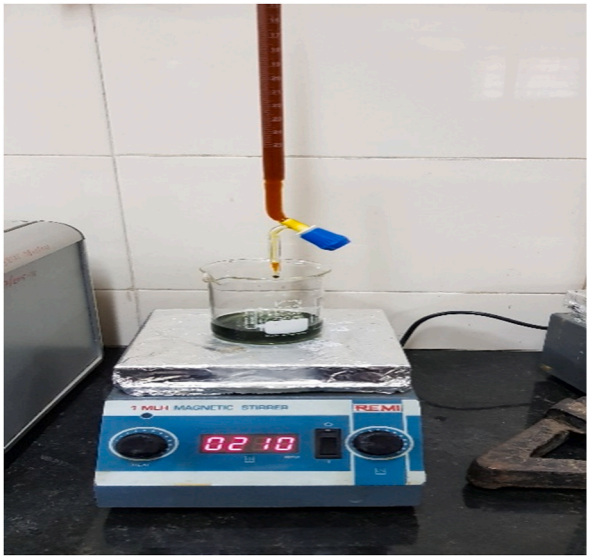

Fig. 2. Experimental setup for the formation of pallets.

type, size, and amount of filler material, as well as the method of synthesis. By optimizing the composition of PPy composites, they can be effectively utilized in various sensor applications, such as gas sensors, bio sensors, and humidity sensors [33].

Polypyrrole (PPy) composites have shown potential for use in gas sensing and detection due to their high electrical conductivity, good stability, and tenable properties. The gas sensing properties of PPy composites can be improved by incorporating various filler materials, such as nanoparticles, carbon nanotubes, and graphene. These fillers can increase the specific surface area, enhance the conductivity, and modulate the electrical properties of PPy, leading to improved gas sensing performance [34].

PPy composites have been used for the detection of various gases, including oxygen, nitrogen oxides, carbon monoxide, and volatile organic compounds (VOCs). For example, PPy composites with graphene or carbon nanotubes as fillers have shown high sensitivity and selectivity toward the detection of oxygen and nitrogen oxides. PPy composites with metal oxide nanoparticles have been utilized for the detection of VOCs, due to their high adsorption capacity and strong interaction with the target gases. In gas sensing applications, PPy composites are typically used as the active layer in sensors, and their electrical resistance changes in response to the presence of target gases. This change in resistance can be correlated to the concentration of target gases, allowing for quantitative gas sensing and detection. Overall, the use of PPy composites in gas sensing and detection offers a promising alternative to conventional gas sensors, due to their unique combination of electrical conductivity, stability, and tunable properties.

Polypyrrole (PPy) composites have shown potential for use as volatile organic solvent (VOS) sensors due to their high electrical conductivity and tunable properties. The sensitivity and selectivity of PPy-based VOS sensors can be improved by incorporating various filler materials, such as metal oxide nanoparticles, carbon nanotubes, and graphene. These fillers can increase the specific surface area, enhance the conductivity, and modulate the electrical properties of PPy, leading to improved VOS sensing performance. PPy composites with metal oxide nanoparticles have been reported to have high sensitivity and selectivity towards the detection of various VOSs, including ammonia. The high adsorption capacity and strong interaction between the metal oxide nanoparticles and ammonia result in a significant change in electrical resistance of the PPy composite, allowing for quantitative sensing of ammonia [35]. In VOS sensing applications, PPy composites are typically used as the active layer in sensors, and their electrical resistance changes in response to the presence of target VOSs. This change in resistance can be correlated to the concentration of target VOSs, allowing for quantitative sensing and detection. Overall, the use of PPy composites in VOS sensing and detection offers a promising alternative to conventional VOS sensors, due to their unique combination of electrical conductivity, stability, and tunable properties. The high sensitivity and selectivity of PPy-based sensors towards ammonia make them a promising choice for sensing applications in industrial and environmental monitoring [36–38].

Although, an electronic component have become smaller and easier to construct, as well as more responsive, due to advancements in small-scale manufacturing, miniature design, and fabrication/assembly offered by electrochemical techniques, fast bio-sensing, and immuno-sensing, information-gathering systems that can be conveniently incorporated into technology, such as, microprocessor-based electronics, have become necessary in recent years. Biosensors, immunosensors [39], and biomimetic affinity sensors are necessary for these applications since they can be produced in large quantities. Biological recognition elements in close proximity to an appropriate transducer, which may translate a biological recognition reaction, or eventually the biocatalytic process into a quantifiable electronic signal, are the basic components of biosensors and immunosensors. Due to its excellent biocompatibility and simplicity in immobilizing a variety of physiologically active chemicals, Polypyrrole is mostly employed in biosensors, and immunosensors [40,41].

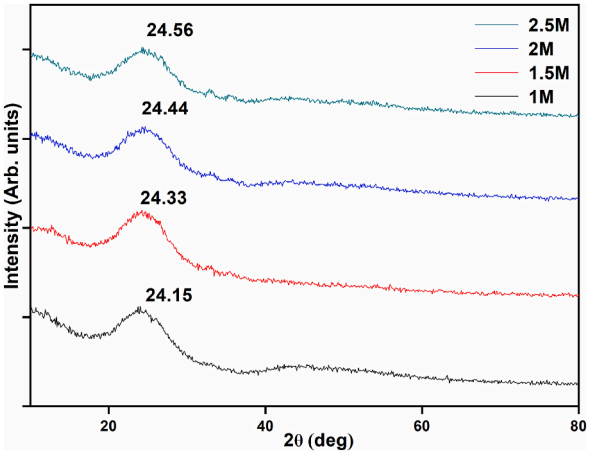

Fig. 3. X-ray diffraction pattern for Polypyrrole produced using $FeCl_3$.

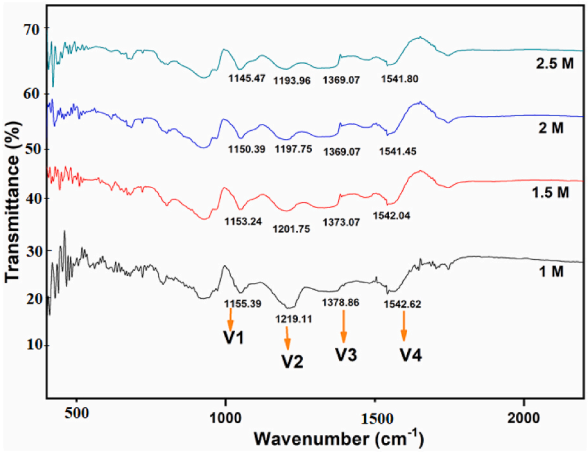

Fig. 4. FTIR spectra of synthetic Ppy samples for oxidants.

2. Experimental and procedure

Synthesis of Polypyrrole (Ppy) 0.5 M (1.73 ml) of pyrrole (monomer) was taken in 50 ml of distilled water, also surfactant CTAB of 0.025 M (0.455 g) was added in the same solution. Oxidant $FeCl_3$ of different concentrations (1 M, 1.5 M, 2 M, and 2.5 M) was added in 50 ml of distilled water. The pyrrole solution was kept on a magnetic stirrer and the FeCl3 solution was added slowly by using a burette. Polymerization was done for 4 h at room temperature. At the end of the reaction final product, Ppy was filtered, washed with distilled water, and dried for about 12 h at 60 °C. Four Ppy samples were prepared of different concentrations of FeCl3, while the concentration of pyrrole and surfactant was kept the same. The final product was black colored powder. The experimental arrangement is shown in Fig. 2. Ppy powders were compressed uniaxially for the formation of a pellet of diameter 12 mm and thickness about 3 mm, then $20^\circ < 2\theta < 25^\circ$ revealed that the resulting Polypyrrole powders [42] were amorphous in nature. Such a broad peak usually indicates a short-range arrangement of chains. The diffraction peaks centered at around 24° shows some displacement samples were characterized by using XRD, FTIR, and SEM. The conductivity of samples was also measured. Chemical reaction of polymerization of pyrrole by using FeCl3 is,

$$nC_4H_2NH + 2nFeCl_3 \rightarrow (C_4H_2NH)_n + 2nFeCl_2 + 2nHCl$$

3. Result and discussion

The X-ray diffraction pattern for Polypyrrole produced using $FeCl_3$ as an oxidant is shown in Fig. 3. Broad peaks can be seen in the area in the XRD patterns for four samples employing various oxidant concentrations [43–45], on further analysis of $FeCl_3$ at a concentration of 1–2.5 M. The peaks are often shifted toward the higher angle by about 0.5 when an oxidant is added. This suggests that the interplanar gap diminishes when oxidant is added.

Table 1
FTIR frequency bond variation.

Frequency (ν) (cm^{-1})	Bond
ν1 (1100)	C–C stretching
ν 2 (1200)	C–H bond
ν 3 (1400)	C–N bond
ν 4 (1600)	C═C stretching

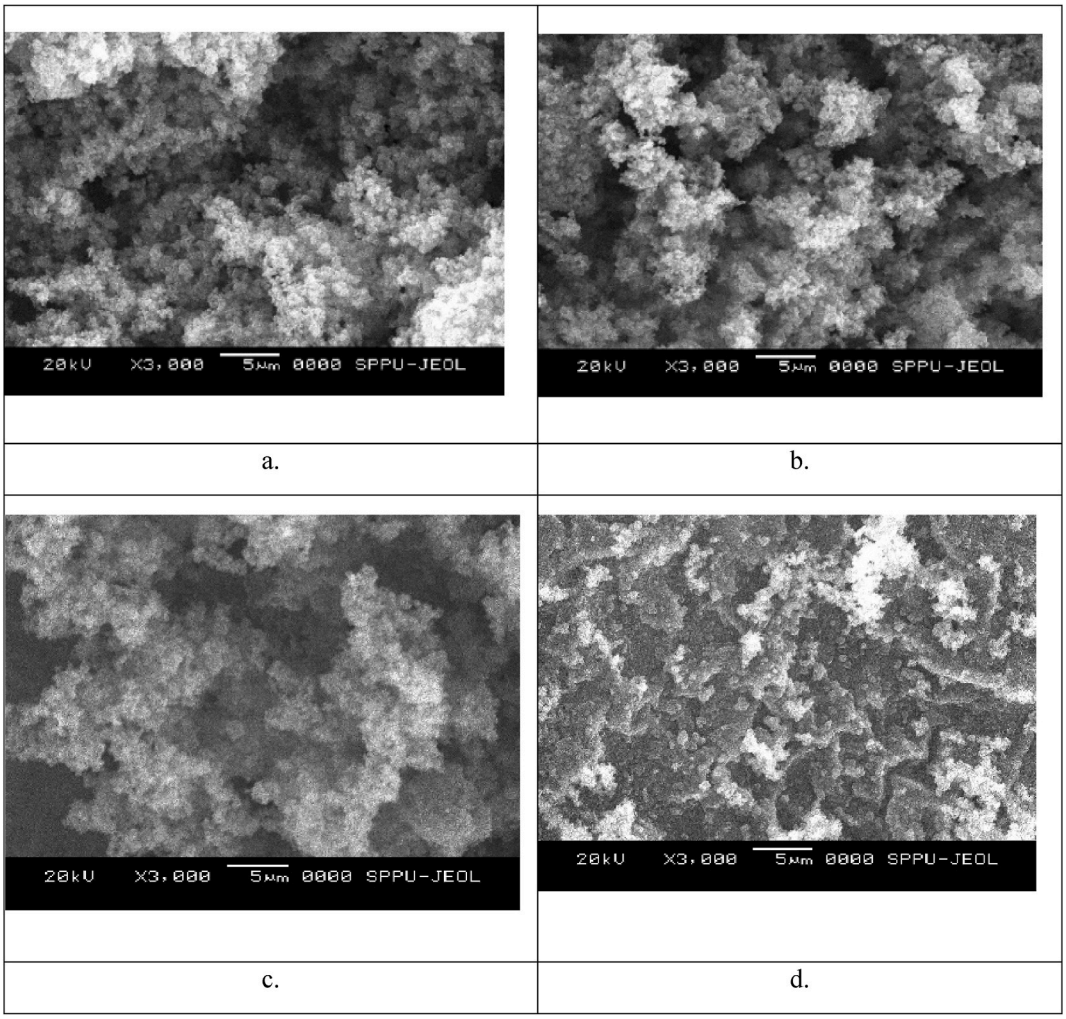

Fig. 5 (a–d). SEM images with different concentrations of oxidizing agent.

To validate the polymerization of the monomer to the polymer during the synthesis process, FTIR examinations were conducted. Fig. 4 displays the ferric chloride FTIR spectra of synthetic Ppy samples for oxidants. To prove polymerization [46], the FTIR spectra of the Ppy samples were taken in the 4000 to 500 cm^{-1} regions. Peaks at 1400 cm^{-1} and 1600 cm^{-1} are typical peaks that correlate to the C═C stretching, respectively. The peak at 1100 cm^{-1} is caused by C–C stretching, while 1200 cm^{-1} represents, respectively, the C–N, and C–H planar deformation bonds. The bond vibrations for the associated frequency are shown in Table 1.

Fig. 5(a–d) illustrates the microstructure of Polypyrrole samples for different concentrations of ferric chloride as oxidizing agents while py and surfactant concentration was kept constant. All samples show porous nature. The porous nature observed in SEM images is suitable for gas sensing. And the average size of the globules produced during FeCl3 polymerization was found to be 0.25 m. Individual granules were almost spherical, and closely packed when they were viewed. Such spherulites appear to be stacking one on top of the other and forming a continuous structure. Furthermore, the morphological trait was spongy in nature, making it difficult to tell the granules apart from one another. This demonstrates that a densely packed structure is created [47,48], supporting our earlier finding based on X-ray diffraction. The ultimate properties, particularly conductivity, are significantly influenced by the

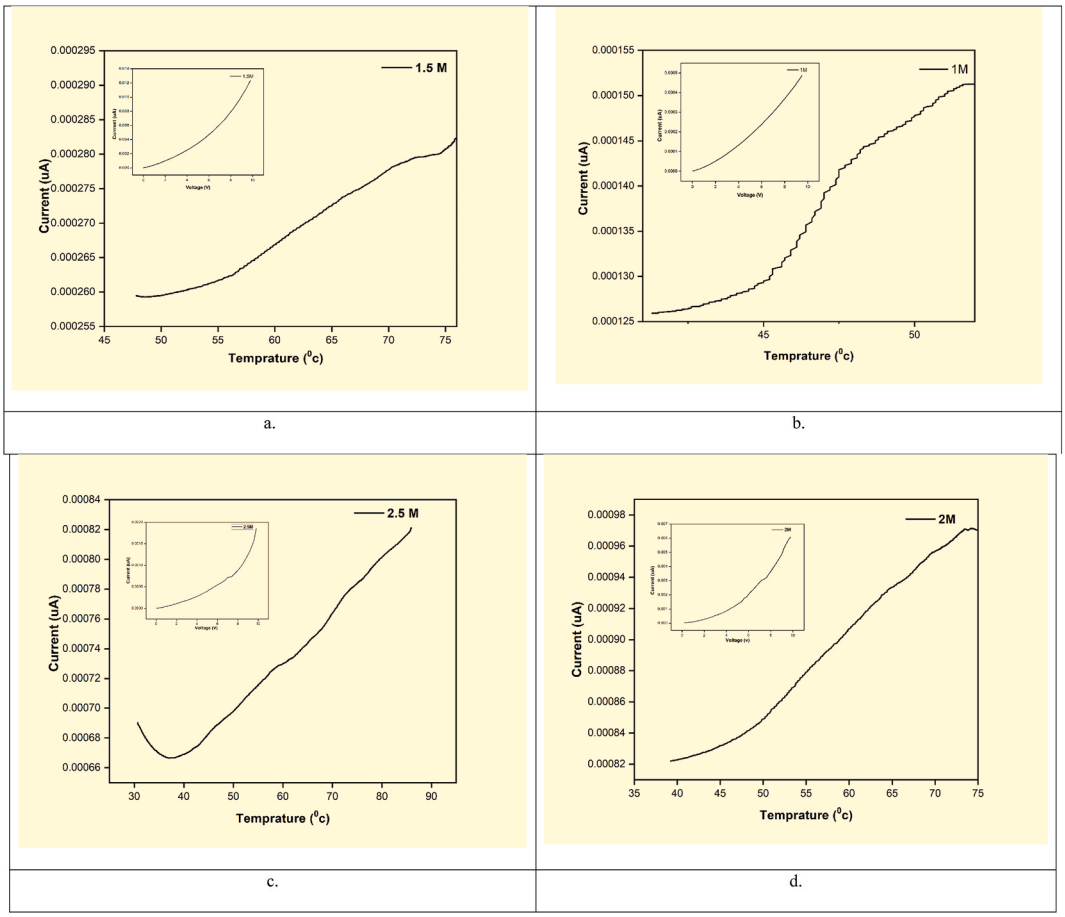

Fig. 6(a–d). I–V characteristics of Ppy (FeCl3 concentration of 1 M, 1.5 M, 2 M, and 2.5 M respectively).

Table 2
Conductivity for different concentration.

Ratio of O/py	Conductivity (ohm^{-1} cm^{-1})
2	4.07×10^{-5}
3	4.46×10^{-4}
4	4.77×10^{-4}
5	8.49×10^{-5}

morphological structure of the Ppy samples.

Ppy samples' electrical conductivity was tested using the two-probe method. The conductive nature of Ppy was confirmed by studying I–V characteristics. Up to 2 M, the conductivity of Ppy increases as the concentration of $FeCl_3$ rises, after an excess increase in the concentration of $FeCl_3$ leads to a decrease in conductivity. This might be attributable to the development of carboxyl flaws brought on by excessive $FeCl_3$ oxidation. Typically, carbonyl defects might disrupt conjugation, which would obstruct charge transfer and subsequently diminish conductivity [49]. This could be the result of overoxidation. The table shows the change in conductivity for different O/py ratios. The exponential nature of the curve shows the semiconductor nature of Polypyrrole. Ppy is a P-type semiconductor. Fig. 6(a–d) shows the I–V curve for different concentrations of oxidant in terms of current and temperature. Table 2 shows conductivity increasing up to O/py = 4 after this ratio, there is a decrease in conductivity.

The gas sensing of most conductive Polypyrrole samples of ratio O/py = 4 ($FeCl_3$ = 2 M, Py = 0.5 M) was carried out using a gas sensitivity measurement setup for gas NH_3. Ammonia gas is widely utilized in a variety of sectors, including the chemical sector, which manufactures nitric acid, the petrochemical sector, industrial hygiene monitors, and calibration gas mixes for environmental emission monitors [50]. Ammonia gas is also used in the production of plastics, explosives, textiles, insecticides, dyes, and other products [51–53]. It is frequently utilized in the production of silicon nitride and gallium nitride (GaN) in the electronics industry. In some refrigerators, it serves as the refrigerant in place of freons. Ammonia, however, belongs to a class of extremely dangerous gases. Ammonia inhalation can lead to respiratory system abnormalities as well as eye discomfort. According to the preceding description,

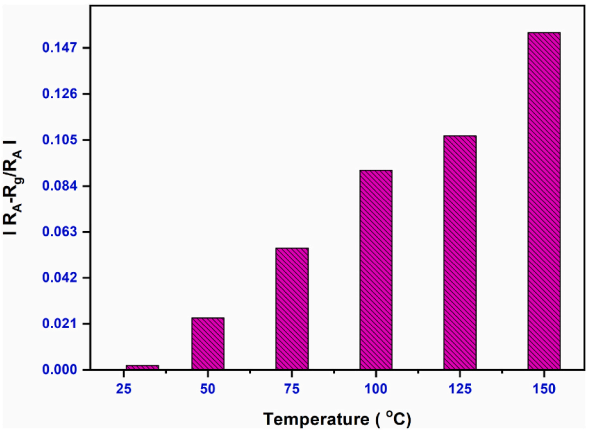

Fig. 7. Change of conductivity with temperature variation.

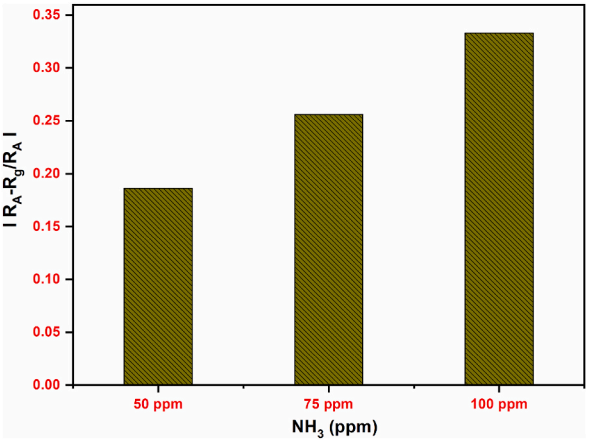

Fig. 8. Effect on Sensitivity with an increase of ppm of NH_3.

sensors that can detect ammonia at very low concentrations while operating at room temperature are essential [54–56]. That's why Ppy was used. The measurements were performed for two different cases. In the first case, the temperature was varied between 30 °C and 150 °C with a step of 25 °C. The sample was placed inside the chamber on a heater, provided with ohmic contacts, and then exposed to a 10 ppm concentration of NH_3. Using a Keithley 2400 source meter, the change in sample resistance following gas exposure was documented [57–59]. In the second case, the gas concentration was changed and sensitivity was measured at room temperature. The concentration of NH_3 was changed by step 25 ppm between ranges 50 ppm to 100 ppm.

As a surface-controlled phenomenon, oxygen adsorption, lattice defects, grain size, surface area, and pore size all play significant roles in the gas-sensing mechanism [60–62]. The temperature has a significant impact on gas sensitivity. Since the Ppy is a p-type semiconductor there is an increase in conductivity as temperature increases [63–65]. Fig. 7 shows sensitivity vs temperature, in this case, the concentration of NH_3, was kept at 10 ppm for all temperatures. As ammonia is a reducing gas, it will donate electrons to the Ppy sample. Ppy is a p-type semiconductor, majority of carriers in Ppy are holes [66–68]. As there is an increase in temperature more electrons will be available at the conduction band, and external electrons from ammonia will also raise the availability of electrons. Electron hole pairing will be done and the resistance of Ppy will increase also sensitivity will increase [69–71]. That's why at higher temperatures sensitivity will be high. Higher sensitivity was measured at 150 °C. Ppy is stable up to a temperature of 150 °C that's why sensitivity was measured up to 150 °C.

In the second case, the concentration of Ammonia was changed and the temperature was the same for all measurements as shown in Fig. 8. Reducing the nature of NH_3 will increase electron concentration at the surface of the pellet [72–74]. As ppm will increase the concentration of electrons will also increase, holes will combine with electrons and resistance will increase [75–77]. That is the reason 100 ppm shows maximum sensitivity.

4. Conclusions

Ppy samples were successfully synthesized using the Chemical Oxidative Method. It was discovered that the electrical, and thermal

characteristics of the generated Ppy samples were significantly impacted by the use of ferric chloride ($FeCl_3$) as an oxidant. Comparably, the impacts of $FeCl_3$, and pyrrole monomer concentrations on the electrical, and thermal stability were investigated, and the results demonstrated that raising the concentration of $FeCl_3$ up to 2 M decreases sample resistivity. It was discovered that getting better conductivity is significantly influenced by the sample's morphological characteristics. Results from FTIR, XRD, and SEM have demonstrated how Polypyrrole polymer forms during synthesis. Gas sensing of Ammonia gas was successfully done for most conductive Polypyrrole samples.

Author contribution statement

Alok Jain, Ansari Novman Nabeel, Sunita Bhagwat, Rajeev Kumar, Rajesh Singh: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper. Shubham Sharma, Abhinav Kumar: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper. Dražan Kozak, Anica Hunjet: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declarations

There is no potential conflict of interest reported by the authors.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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