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Development of Biomaterial based Ammonia gas Sensor

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Abstract— **A comprehensive study on electrical characteristics of garlic membrane along with its application potential as ammonia gas sensor has been presented in this paper. The material potential of garlic membrane, collected from the peel of natural garlic, has been investigated to fabricate a gas sensor. The reversible structural changes are found to be associated with the membrane under exposure of ammonia gas and can be measured as a short duration electrical signal. Sensitivity of garlic membrane towards humidity, temperature and external factors has also been studied. The same has been exploited towards development of a bio-material based potentiometric sensor of ammonia gas with fast response and recovery time.**

Keywords— *gas sensor; garlic membrane; response voltage; response time; recovery time;*

I. INTRODUCTION

Gas sensor is a device which detects gas by the generation of an electrical signal across it. The signal thus generated is due to some temporary change in electrical, magnetic or chemical property of the sensing material under exposure to the stimulus gas. Many inorganic, organic and polymeric gas sensors have been developed so far, among which some are made of toxic materials [1-3], some are with less usage cycle and above all, most of the sensors are expensive. Biomaterials obtained directly from natural materials, can be used as sensing materials by exploiting their sensitivity to environmental parameters namely humidity, presence of gases like ammonia, carbon dioxide etc. Biomaterial based sensors may be supposed to be superior over conventional types in regard to their eco-friendliness biodegradability and cost effectiveness. It has been observed that biological membranous substances can play a better role as gas sensor in the mentioned direction.

Biological membranes can best be described as a selectively permeable material, it means that some ions can pass through and some cannot. Biologists wondered for many years about the nature of barrier that separates cells from their environment. Plant membrane is a barrier to ion movement, and existence of ion channels [4] has been found in it. Ion channels are the aqueous pores through which the ion movement can take place. The direction of the ion movement is being determined by the electro chemical potential gradient. It has been found that electric potential developed across the membrane i.e. membrane potential is of the order of few tens of mV (mili Volt). The nature of the developed potential can be explained by using Nernst equation [4].

Garlic is a medicinal plant. Garlic root is a compound bulb, consisting of several cloves enveloped by a skin or membrane, rich in Sulphur and Iodine. A garlic bulb has a pungent odour and an acid flavor due to the presence of Thiol (S-H) group. Its scientific name is *Allium sativum* and it belongs to the family of *Liliaceae/Alliaceae.* Electron energy loss spectroscopy [5] shows that its clove and membrane, can absorb high amount of Cadmium (Cd), which is used in purification of Cd contained organic specimens. The study of electrical properties of Garlic clove is important in many respects including Lipid replacement therapy [6]. The membrane of garlic contains bio catalyzer [7] in it as cell suspension or in the form of a tissue slice.

The garlic membrane is comprised of Pectin (11-12%) as characteristic constituent [8] and its extract contains six Phenyl propanoids [9]. The later compounds are isolated as well as their scavenging effect towards radical l, 1diPhenyl 2 Picryl Hydrazyl has been reported [9]. It has been concluded [9] that those components of garlic skins show anti oxidant effect. Presence of N-trans-Coumaroyloctopamine, N-trans-feruloyloctopamine etc. were also identified in the membrane [9].

The garlic membrane exhibits some reversible structural changes, on exposure to ammonia gas, which are detectable in the infra red region of its transmission spectrum. The short duration electrical signal, generated due to momentary structural change, was amplified using simple electronic circuit and recorded potentiometrically. The signal voltage, thus generated, tends to decay while the membrane released the adsorbed gas. This is the underlying principle of working of the present sensor of ammonia gas. In practice the membrane is sandwiched between redox electrode pair. Typically it absorbs gas and releases the gas within a short time interval. This material characteristic may be exploited in designing a good gas sensor.

The garlic membrane has a complex bio-molecular structure. It exhibits variation in direct current (DC) conductivity with change in temperature. Exposure to ammonia gas the membrane also exhibits variation of its electrical conductivity at a particular temperature. The temperature variation of electrical conductivity and the sensitivity of the later towards external influence, like humidity and ammonia gas have been studied in this work. A comprehensive study on electrical characteristics of garlic membrane along with its application potential as ammonia gas sensor has been presented in this paper. The sensor thus designed with garlic membrane as transducer, is of low cost profile, nontoxic and eco-friendly. The details of the study, results, analysis and conclusion therein are given in the following sections.

II. MATERIAL AND METHODS

A. Material

The material under investigation is fresh peel of garlic which is known to be garlic membrane with average thickness 25-30μm. Seven successive outer layers of a garlic clove had been isolated, washed with distilled water and allowed for adequate drying. It was shaped properly to fit in between the redox electrode pair. The dried layers were then sandwiched between redox pair of electrodes, one of which was blocking type anode and the other one was porous cathode, to form an experimental proto-cell.

B. Electrical Measurements

The variation of developed voltage, across the terminals of the proto-cell on exposure to ammonia, known as the response voltage, was recorded with temperature as parameter. The amplitude of signal voltage generated across the membrane on gas exposure was found to be low (~mV). It was further amplified by a simple Operational Amplifier (Op-Amp) based circuit (Fig. 2).

The said measurement was carried out between temperatures 7ºC to 30ºC at an effective constant humidity level. Response time, the time required to reach at the maximum of response voltage level under exposure of gas and recovery time , the time required to recover the near initial voltage level had also been measured (Fig. 1) between mentioned temperature range.

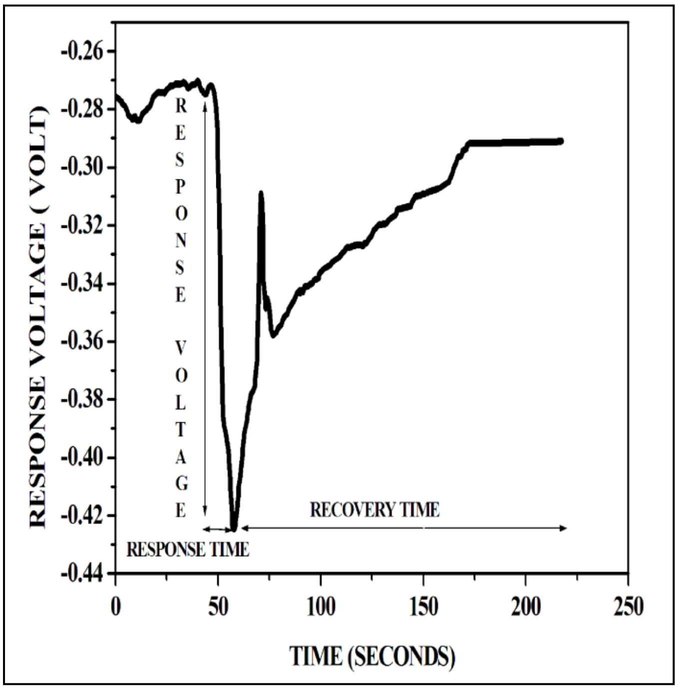


Fig. 1. Variation in Voltage level across membrane cell with time (on exposure to ammonia gas)

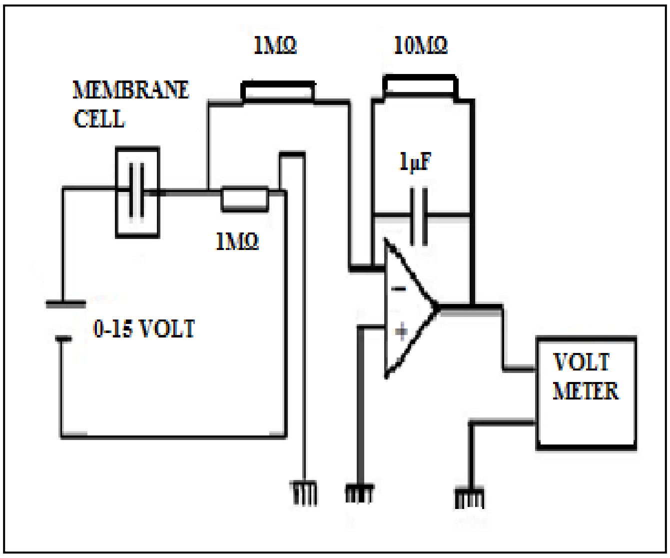


Fig. 2. Circuit layout for amplification of response signal voltage

Adequate amount of ammonia was introduced in the gas chamber so as to reach the gas concentration level in the chamber to10 part per million (ppm). The ammonia gas concentration in the chamber was measured by ammonia detector (Tinsel gas leak detector) with background compensation.

The same technique had been used in studying the response and recovery of all the individual membrane layers. The mentioned recording was also extended to reversal of the membrane surface towards the exposure of the gas.

The variation of cell current with applied potential difference was recorded in the form of Volt-Ampere (V-I) characteristics using Keithley 2400 Source meter. In fact the variation of bulk conductivity was estimated with temperature as parameter in presence and in absence of ammonia (Fig. 3).

C. Fourier Transform Infrared (FTIR) Spectroscopy

A gas sensing material suffers subtle but detectable and mostly recoverable chemical changes in its structure on exposure of stimulus gas. FTIR absorption spectrum of the membrane was studied using FTIR spectroscopy (Shimadzu Model IR Affinity 1 Japan) in compensated air background between wave number 350-7800 cm^{-1}. FTIR study is important to investigate the effect of humidity or ammonia on the internal structure and dynamics of molecular constituent of garlic membrane.

D. Realisation of Sensor

Different types of sensors may be fabricated depending on the nature of response towards the external stimulus. The external stimulus may cause a change in bulk resistivity or conductivity or capacitance or in overall dielectric property in the sensing material along with some detectable and reversible changes in molecular structure. The mentioned changes generate short duration electronic signal, which may be amplified through proper electronic instrumentation (shown in Fig. 2). Measurement of response voltage and recovery time was performed to establish the potential of the material as ammonia gas sensor.

E. Electronic Circuit

The proto-cell containing the membrane specimen in the mentioned set up was connected to a simple DC circuit (Fig. 2) followed by a resistor of Mega ohm (MΩ) order in series. The output across the resistor was further amplified through an Op-Amp based circuit. The capacitor (1 µF) in the feedback path was used to retard the rapidly generated signal in the proto-cell due to exposure to ammonia gas.

III. RESULTS

A. V-I (Voltage-Current) Characteristics

DC V-I characteristics of the membrane was recorded at temperatures 22.6ºC, 25.6ºC and28ºC. The recording was carried out at normal condition and with exposure of ammonia respectively. Fig. 3 shows the V-I plots with temperature as a parameter and are found to be nonlinear in nature.

The DC conductance thus estimated was found to decrease by 6.31% at 28ºC under ammonia exposure. It was due to immediate formation of weak intermolecular hydrogen bonds [10] between surface molecules of membrane and ammonia gas molecules. Presence of hydrogen bonds caused reduction in mobility of the charges and the membrane suffered a loss in conductivity on addition of ammonia. It can be followed that both response and recovery time show nonlinear increase with increase in temperature (Fig. 5(a) and (b)). At 28ºC both the outcomes were found to be larger compared to that at lower temperatures. Longer response and recovery time indicates the decrease in charge mobility across the membrane on exposure to ammonia gas.

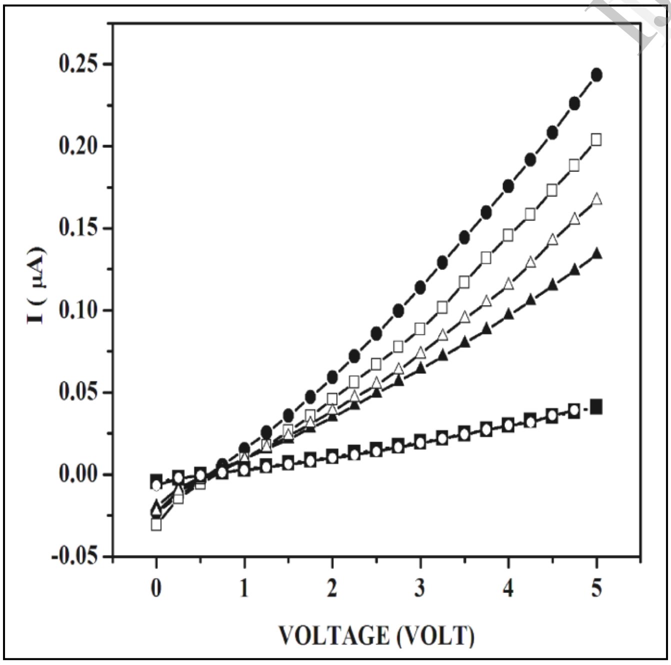


Fig. 3. Study of DC Conductance: Solid circle (●): without ammonia condition at 28^{0}C; Open square (□): with ammonia condition at 28^{0}C; Solid triangle (▲): without ammonia condition at 25.6^{0}C; Open triangle (△): with ammonia condition at 25.6^{0}C; Solid square (■): without ammonia at 22.6^{0}C; Open circle (○): with ammonia at 22.6^{0}C

The observed increase in membrane conductance with gas exposure at 25.6ºC was found to be 38.69% over the corresponding normal state. The observed response voltage at 25.6 ºC was also found to be comparatively higher than those at 22.6 ºC and 28 ºC (Fig. 4). Response and recovery times were found to be appreciably lower (Fig. 5(a) and (b)) at 25.6ºC compared to that at 28ºC. Faster formation of temporary dipoles (faster than formation of hydrogen bond) between gas and surface molecules of the membrane perhaps took place which increased the conductance of the membrane. Such temporary dipole formation is faster in time and weaker in strength compared to that of hydrogen bond. The phenomenon is associated to London dispersion force phenomenon [11]. The presence of large molecules in the garlic membrane may enhance the probability of enhancement of mentioned dispersion force to act.

At temperature 22.6 ºC, V-I plots (under the conditions: with and without ammonia exposure) in Fig.3 are found to be overlapped. The response voltage assumes a minimum value at 22.6 ºC (Fig. 4).

Figure 3 summarized the overall bulk conductance of the membrane rises about 75% due to increase in temperature from 25 ºC to 28 ºC.

The environment plays an important role to the dynamics of the bio-molecular system. The overall nature of response of the biomaterial like garlic membrane remains substantial under normal seasonal variation of environment regarding gas sensitivity.

B. Respone Voltage vs Temperature

Fig. 4 shows the variation of response voltage across the proto-cell due to exposure of ammonia at temperatures between 7 to 28ºC. Response voltage was found to be appreciable within 10 to 15ºC in Fig. 4.

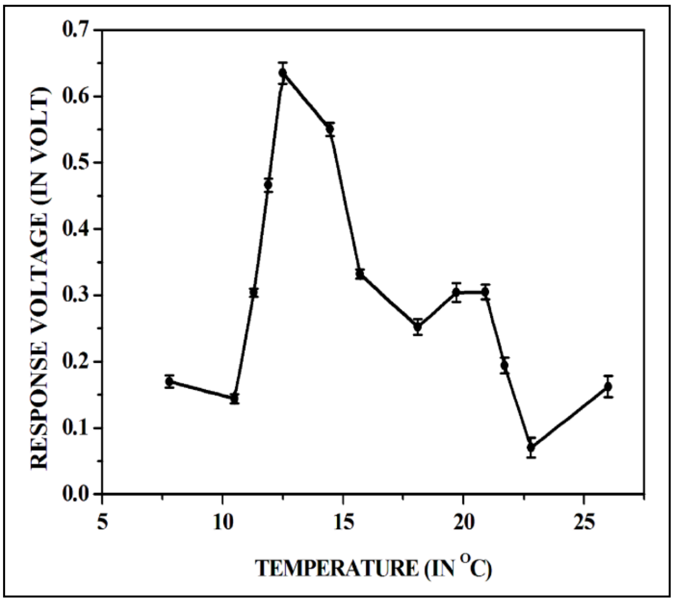
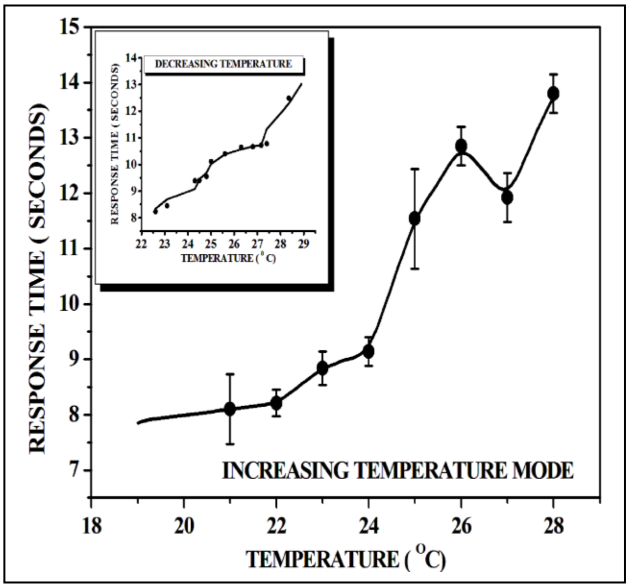
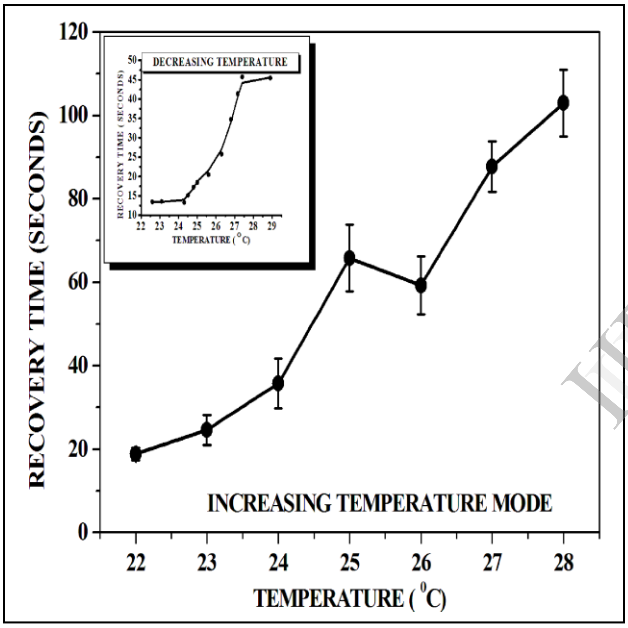


Fig. 4. Mean graph of Response voltage vs. temperature; in the range 7-28^{0}C (Error bar estimated from Standard deviation of five independent measurements)



(a)



(b)

Fig. 5. Mean graph of **(a)** Response time vs. temperature; **(b)** Recovery time vs. temperature (Error bar estimated from Standard deviation of five independent measurements)

C. Response time vs Temperature

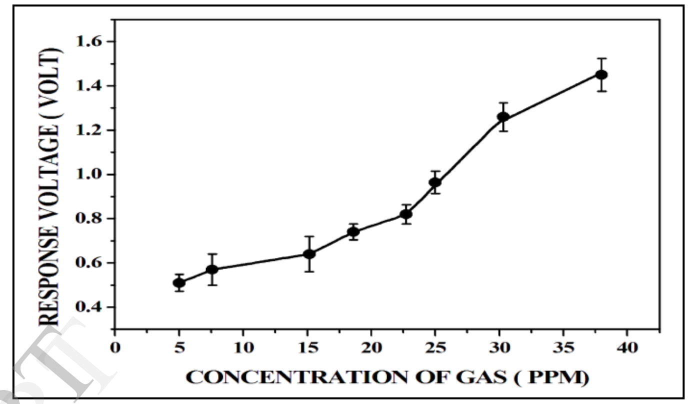
Fig. 5(a) shows an overall increase in response time for both increasing and decreasing temperature mode within the range 22ºC to 28ºC.The said variation has been observed for decreasing temperature mode (inset) and increasing temperature mode.

The inset graph (Fig. 5(a)) for decreasing temperature mode shows, comparatively faster response than that of increasing mode between 24ºC to 27ºC. This signifies that, there was a tendency to form temporary dipoles among the surface molecules of the membrane under the exposure of ammonia gas. This dominates over the formation mechanism of inter molecular hydrogen bonds in case of decreasing temperature mode.

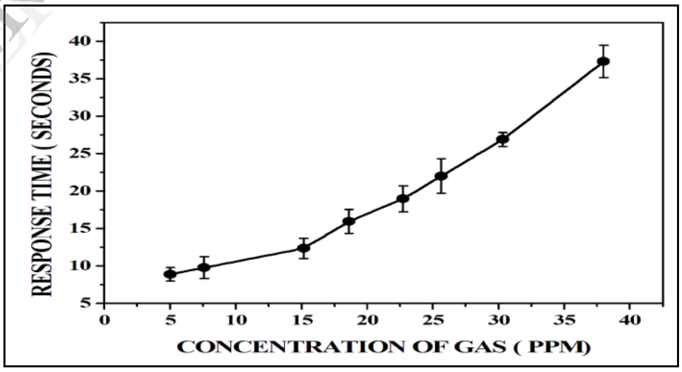
D. Variation in Response Property with variation in concentration of exposed Ammonia gas

Fig. 6(a) shows the variation in response voltage with concentration of ammonia at constant temperature 24^{0}C. The curve shows that the response voltage increases with the increase in the concentration of exposed ammonia towards the membrane. The nature of the graphs in Fig. 6 (b) and 6(c) are showing respective increase in response and recovery time with increase in concentration of gas.

All the three graphs in Fig. 6 are showing approximate linear nature. The concentration of gas can be estimated form Fig. 6(a) for any response voltage. Increase in the concentration of exposed gas to the membrane makes more molecules in the membrane to take part in sensing the gas, which results longer response and recovery time.



(a)



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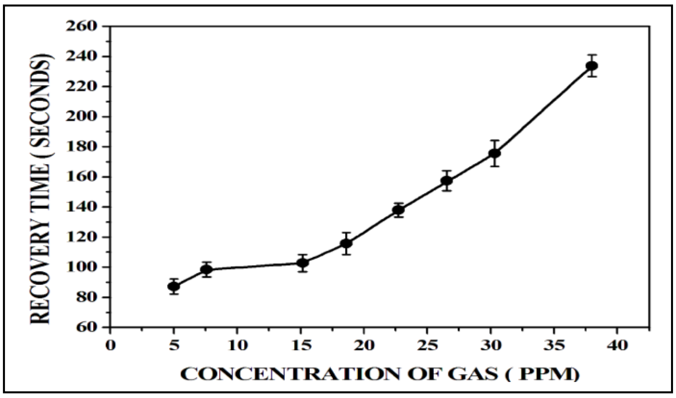


(c)

Fig.6. Mean graph of (a) concentration of gas vs. response voltage; (b) concentration of gas vs. response time; (c) concentration of gas vs. recovery time (Error bar estimated from Standard deviation of five independent measurements)

E. FTIR Results: Layer wise study

FTIR transmittance spectrum in the finger print region of seven successive layers, collected from the surrounding of the garlic clove, shows distinct structural difference from layer to layer (Fig. 7).

The finger print region carries both the information of bond stretching and bending vibration modes of different functional groups. It is easy to observe the marked variation in layer wise transmittance and peaks at different wave numbers in the said region. Presence of hydrogen bond also makes difference in layer wise chemical behavior. Variation in the number of hydrogen bonds causes difference in IR spectrum of successive layers.

Fig. 7 shows the layer wise FTIR spectrum, with notable structural difference. The difference in the structure also creates difference in the response towards ammonia gas along with response and recovery time. Multiple combination bands of medium intensity are observed near 2000-3000 cm^{-1} due to the presence of N-H stretching vibration concerned to Amine group in Fig. 7. The region near 2373-2379 cm^{-1} shows comparatively stronger absorption in layer 2. Significantly this peak is due to C ≡ N stretch related to Nitrile compound [12] in the specimen.

The broad band in Fig. 7 near 3100-3600 cm^{-1} is due to superposition effect of O-H and N-H bond stretching along with hydrogen bonding. It exhibits clear distinction of the distribution of molecules with different functional groups in successive layers. The finger print region below 2000 cm^{-1} shows effect of combination of bond stretching and bending of different chemical groups with detectable difference in layer wise analysis. Characteristic peaks of benzene ring, C=O stretching, C=C stretching etc. can be found in combined form [12] in this region.

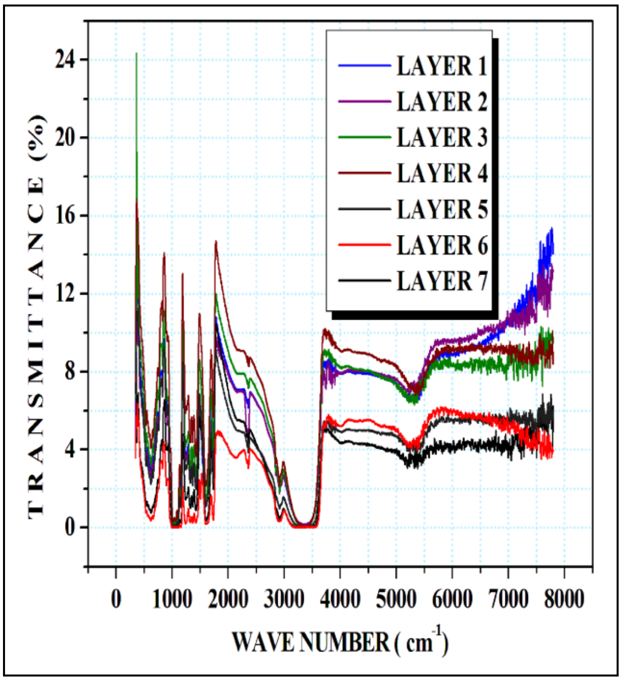


Fig. 7. FTIR spectrum of garlic membrane layers at room temperature

The spectra show high possibility for adsorption of ammonia gas due to the presence of such functional groups. Presence of weak hydrogen bonds supports the temperature sensitivity [10] of the garlic membrane.

F. Humidity Sensing Property

The influence of humidity affects the sensing property of the garlic membrane. Different_parts of the FTIR spectrum of the humid membrane show a marked difference from that of the normal dry membrane. Fig. 8 compares changes in peak positions near 352 cm^{-1}, 359 cm^{-1}, 363 cm^{-1}, 375 cm^{-1} etc. in the FTIR spectrum of the dry garlic membrane (Fig. 8: shown in red). Exposure to excess humidity to garlic membrane results shifts in the mentioned peaks (Fig. 8: shown in blue).

Fig. 8 summarizes the sensitivity of the membrane towards humidity. Humidity also has a high influence on the dielectric property of the garlic membrane. The dynamics of the membrane suffers substantial change in its capacitive impedance and resistance under exposure to humidity [14].

G. Ammonia gas sensing property

Table 1 shows a comparative study of gas sensing performance of either sides of membrane specimen in a layer wise observation. Response time less than or equal to 1 minute is considered to be good, response time within 1-2 minutes is considered to be moderate (marked as 'modrt' in Table 1) and response time greater than or equal to 2 minutes is considered to be 'bad'.

The quality of sensing ammonia gas was found to be different for different layers of garlic membrane. Table 1 shows the layer wise variation of the response of the sensor material due to exposure of ammonia gas. The layers were assigned serial number increasingly from the outermost to the innermost garlic membranes surrounding a clove. Table 1 also compares the response of either sides of membranes and the results show distinct difference.

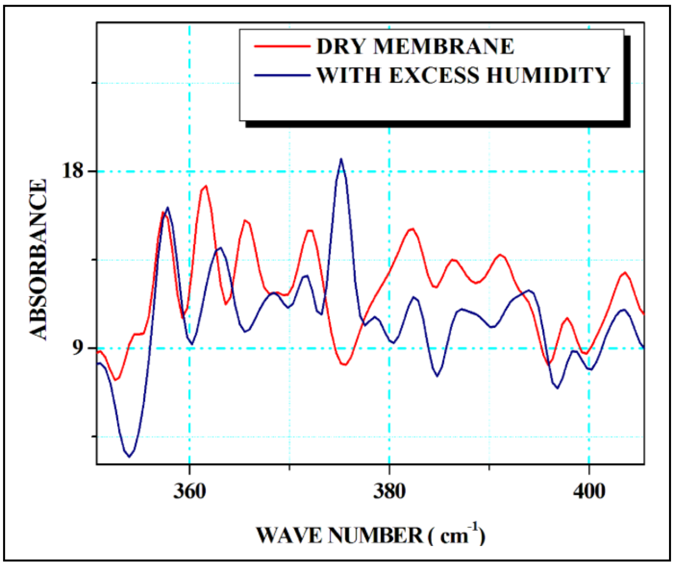


Fig.8. Part of FTIR spectra of garlic membrane to study the effect of humidity

TABLE I.

Layer	Table Column Voltage (Volt)			Response quality	
	Without NH_3	*With NH_3*	*Change*	*Response time*	*Recovery time*
1.a) Concave side	_3.88	_4.14	_0.26	≥1min, modrt	≥2min,bad
b) Convex side	_2.28	_2.47	_0.19	≥1min, modrt	≥2min,bad
2.a) Concave side	_1.67	_1.83	_0.16	≤1min, good	≥1min, modrt
b) Convex side	_0.63	_0.74	_0.11	≤1min, good	≥1min, modrt
3.a) Concave side	_0.77	_0.96	_0.19	≤1min, good	≥2min,bad
b) Convex side	_0.35	_0.48	_0.13	≤1min, good	≥2min,bad
4.a) Concave side	_0.69	_0.92	_0.23	≤1min, good	≥2min,bad
b)Convex side	_1.17	_1.23	_0.06	≥2min,bad	≥2min,bad
5.a) Concave side	_0.79	_0.92	_0.13	≤1min, good	≤1min, good
b) Convex side	_0.77	_0.89	_0.19	≤1min, good	≥1min, modrt
6.a) Concave side	_2.66	_2.83	_0.17	≤1min, good	≤1min, good
b) Convex side	_2.00	_2.16	_0.16	≤1min, good	≤1min, good
7.a) Concave side	_2.69	_2.89	_0.26	≤1min, good	≥1min, modrt
b) Convex side	_1.10	_1.18	_0.08	≥1min, modrt	≥2min,bad

Supply Voltage 3 Volt;Room temperature 24 ^{0} C, Concentration of ammonia 10 ppm (with background compensation)

H. Layer wise Spectroscopic study of sensing material under exposure of Ammonia gas

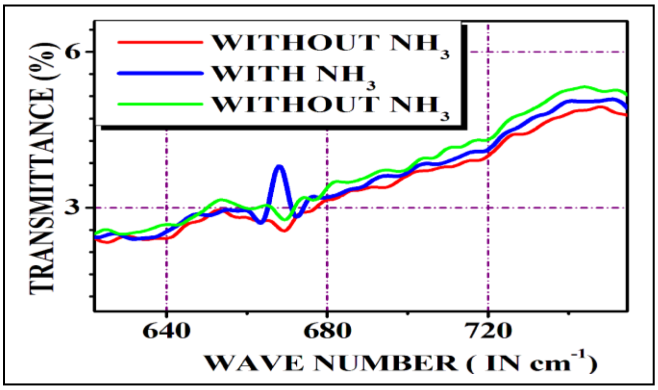
FTIR spectrum of each layer of garlic membrane had been recorded under the condition: (i) without exposing the gas, (ii) exposing the gas to it and (iii) after withdraw of gas. The study was made to investigate the temporary structural change in the membrane due to exposure in ammonia gas. The time lag between steps is 10 minutes.

The temporary influence of ammonia gas on the different layers of garlic membrane has been shown in Fig. 9. The absorption peak near 668 cm^{-1} in Fig. 9 (a), (b) and (c) for layers 4 to 6, corresponds to wagging vibration of NH group of primary Amide [12] of normal membrane under condition (i) (in red). The adsorption of ammonia increased mass of the molecule leading to reduction of the wagging mode of vibration and it results a sudden increase in transmittance. The distinct difference can be observed near 668 cm^{-1} in Fig. 9 (a), (b) and (c) for layers 4 to 6 under measuring conditions (i) (in red) and (ii) (in blue). The initial spectra (in red) of layers 4 to 6 mostly recovered (in blue) under measuring condition (iii) that is 10 minutes after removal of gas. Layers 4 and 6 (Fig. 9 (a) and (c)) show abrupt increase in transmittance at 668 cm^{-1} on ammonia gas exposure, indicating higher absorption of photon energy, which automatically signifies lower hosting time (calculated to be 10^{-9}s) of the gas on the membrane surface, following comparatively faster response and recovery time.

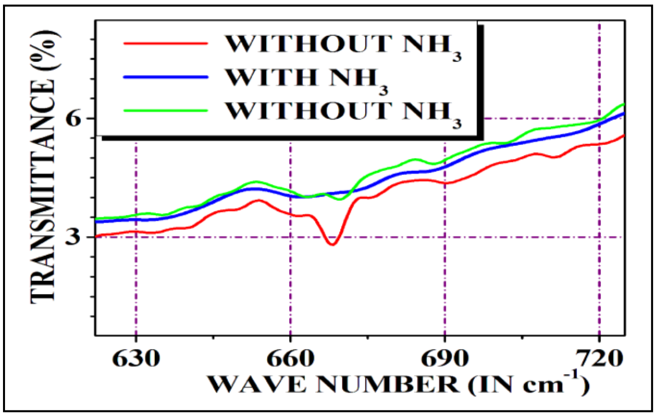
It is also observed for layer 6, that wagging mode of vibration of NH group is lost and a subtle absorption peak of 'in plane bending vibration' reappears near 2360cm^{-1}(Fig. 9(d)). Such shift can take place due to vibration of the said group under temporary hydrogen bonded condition [13]. Same phenomenon is applicable for layer 4 also. The mentioned structural changes are mostly recoverable for layers 4 to 6, after a time lag of 10 minutes of ammonia gas exposure. This phenomenon makes the garlic membrane as a good sensor of ammonia gas. An absorption peak (in red) (Fig. 9(b)) near 668 cm^{-1}, which appeared before exposure of the gas, disappeared after exposure of ammonia to layer 5. The peak again tends to appear (Fig. 9(b)) (in green) after 10 minutes of the exposure of the gas. A permanent change near 2370 cm^{-1} is observed in layer 7, as the absorption peak becomes absent permanently on addition of ammonia (Fig. 9(e), (f)). This 7th layer is adjacent to the inner clove and suffering such permanent change. This proves its poor worth regarding sensitivity to the gas unlike the other layers.

The variation in transmittance with wave number is also leads to the variation of refractive index of the membrane. Using Kramer Kronig transformation refractive index vs. wave number for the material has been plotted for (i) without, (ii) with and (iii) after withdrawal of ammonia gas exposure in Fig. 10.

Fig. 10 shows notable changes in refractive index of the material on exposing ammonia at different wave numbers. The refractive index is found to be partly reversible after withdrawal of ammonia. It occurred due to the presence of the ammonia gas in the surrounding of the membrane within FTIR spectroscope chamber. The inset plot in Figure 10 is a portion of the main plot, which shows a sudden rise in



(a)



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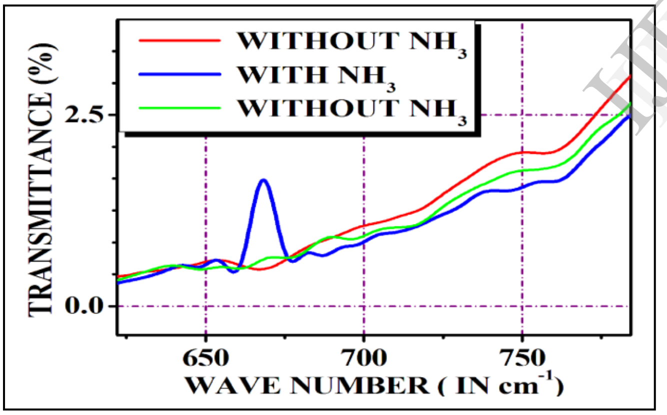

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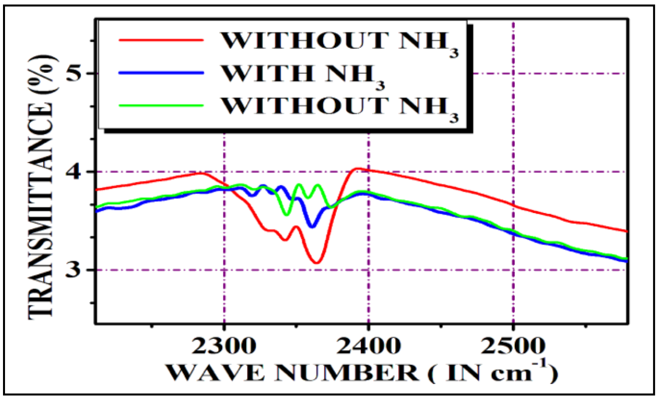

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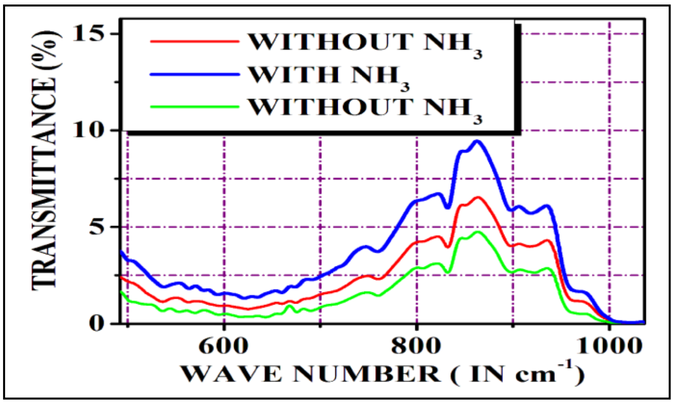
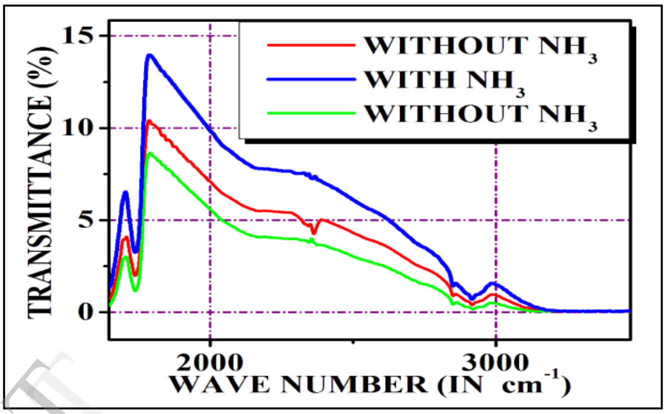


(e)



(f)

Fig. 9. Results of layer wise study on membrane sensing:[red]- without NH_3; [blue]- with NH_3; [green]- without NH_3: (a) Layer 4 (621-754 cm $^{-1}$),(b) Layer 5 (622-725 cm $^{-1}$),(c) Layer 6 (640-800 cm $^{-1}$),(d) Layer 6 (2200-2650 cm $^{-1}$),(e) Layer 7 (491-1036 cm $^{-1}$), (f) Layer 7 (1642-3467 cm $^{-1}$).

refractive index near 668 cm^{-1}. This can be correlated to the rise in transmittance near 668 cm^{-1} on ammonia exposure in Fig. 9(a) and is due to wagging mode of vibration of NH group of primary Amide.

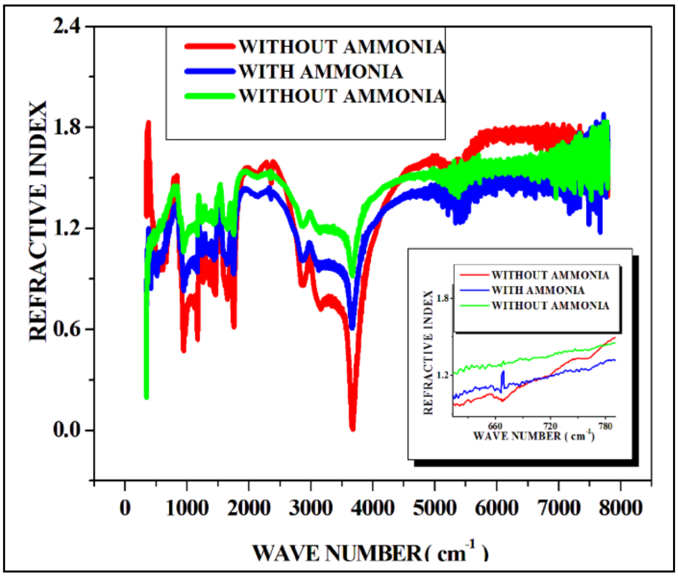


Fig. 10. Refractive Index vs. wave number plot, using Kramer-Kronig transformation of measured transmittance vs. wave number data of FTIR spectra

IV. DISCUSSION

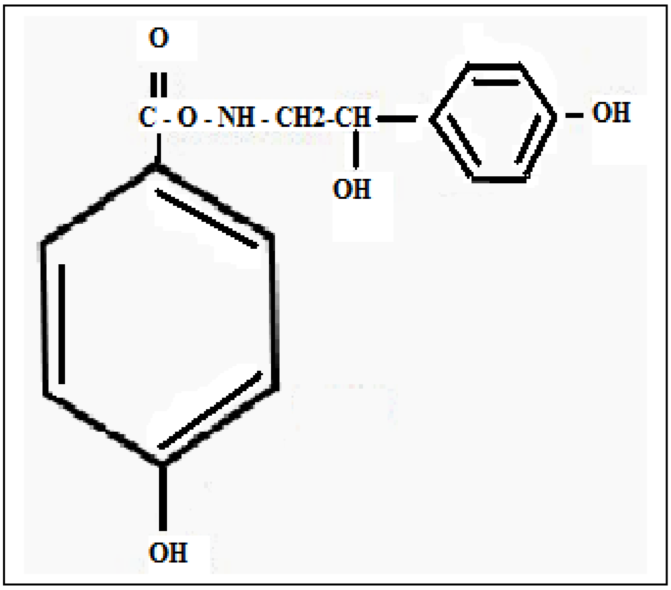
It is clear from the results of present experimental studies, that the garlic membrane could be a good sensing material of ammonia gas with moderate response and recovery time between temperatures 7^0C to 28^0C. Exact recovery of initial voltage level is almost impossible for higher temperatures due to increasing fluctuation in response character associated to molecular dynamics.

There are many hydrogen bond sensitive groups in the garlic membrane structure, which have direct or indirect influence on the quantity of ammonia adsorption. Fatigue in ammonia adsorption was observed near 22^0C and 10-11^0C (Fig. 4(a) and 4(b)) while the gradual increase or decrease in temperature. An increase in concentration of ammonia gas can give a good response voltage (Fig. 6(a)).

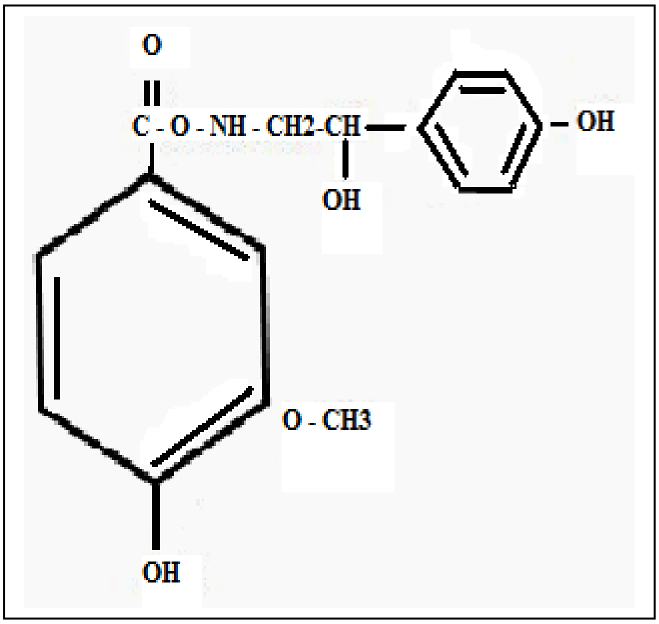
Strong evidence of presence of N-trans-Coumaroyloctopamine, N-trans-feruloyloctopamine [9] and layer wise study (Fig. 7and Fig. 9) may explain such response of the membrane towards ammonia gas.

The chemical structures of Coumaroyloctopamine and feruloyloctopamine [15] in Fig. 11 show that they are large enough to allow temporary inclusion of ammonia gas through transient induced dipole interaction like London dispersion phenomenon. Fig. 10 shows change in dielectric behaviour of the membrane on exposure to ammonia. Linkage of ammonia to different parts of the molecule results an increase in the mass of the portion to reduce the wagging frequency directly or indirectly. This results temporary storage of ammonia increases the membrane potential momentarily and immediate release of the gas brings back the membrane potential to its initial level.

Layer wise FTIR study in Fig. 7 reveals the existence of hydrogen bond, causes temperature sensitivity. V-I plots at different temperatures also reveals the effect of temperature on sensing property of the membrane. The sensitivity towards humidity is clear from Fig. 8.



(a)



(b)

Fig. 11. (a) N-trans-Coumaroyloctopamine, (b) N-trans-feruloyloctopamin

V. CONCLUSION

The complex biological system like garlic membrane shows a regular sensibility towards ammonia gas, humidity and temperature as well. A cost effective ammonia gas sensor of high profile has been developed out of it. The garlic membrane is found to acquire all the qualities of a gas sensor within the given temperature range. Garlic membrane is natural, nontoxic and eco-friendly. Its distinct response towards ammonia gas is exploited to fabricate a gas sensor of low cost profile with the use of simple instrumentation. The developed sensor exhibits good response character with faster response and recovery time.

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