CHAPTER III

SHELLAC WAX ELECTRETS

Introduction

The phenomena of electret formation and the interesting properties exhibited by the forming material during and after formation, have attracted the attention of workers in many laboratories (Appendix I), where the phenomena is described in details along with the associated properties. But no theory has yet been developed which can give a satisfactory explanation of all the observed processes occurring in electret formation. This can be attributed partly to the lack of complete knowledge of the structure and constitution of the material—usually waxes—used in electret preparation, and partly to the lack of thorough and detailed investigations of different characteristics of the electret. The waxes are commercial compounds consisting of a number of organic long-chain compounds like the higher alcohols, acids and esters. The properties of these higher alcohols, acids etc. which constitute the waxes are not completely known even individually and hence it becomes all the more difficult to understand fully their behaviour in waxes, where they may exist either free or in combined state as esters.

Some dielectrics produce fairly strong charges after the application of high voltages, even without heat treatment (1). Electrets can be prepared both with and without melting the wax (2, 3, 4). Thiessen and others could obtain permanent electrification of the dielectric without
heating it, but the value of electrification obtained was considerably low. Gross and Denard found that stable electrets can be obtained without reaching the melting point of the substance, but the field strength obtained was lower than that of electrets prepared with heat treatment. Groetzinger and Kretsch (5) also prepared electrets without melting the wax.

From the results of various workers it can be seen that good electrets can be prepared by applying the field when the substance is in a fluid state, and then allowing it to solidify, with the field on. Best results can be obtained when the polarising field is removed once the wax has thoroughly solidified but still remains at an elevated temperature (6). Tiku found that the resulting charges diminished if the field is removed while the mixture is not yet completely solidified or when the field is applied for too long a period after solidification.

Electrets can be prepared without the application of an external field. Thiessen and others (3) have made electrets by allowing the wax to solidify from its melt, between tin electrodes. The magnitude of charge obtained was approximately 1/4 of the charge on electrets prepared by application of the field. The same authors report that surface charges of a more or less permanent nature, appear even if the mixture is simply rubbed with a hair brush and kept between tin foils without any heating or external field. But this is a surface effect only and not a true electret effect. Good and Stranathan prepared electrets from carnauba wax
and resin mixture under different cooling conditions. They undertook an extensive study by X-ray diffraction methods just after the preparation of electrets and again after the reversal of charge has taken place. They found that electrets prepared by cooling in air showed a clear orientation, as observed by Ewing (8) and Kakiuchi (9). But electrets prepared by cooling in an iron cylinder showed no trace of crystalline orientation inside. The discrepancy so observed in these electrets can be due to different strains produced in the electrets under different cooling conditions.

Parasuramiah and Bhawalkar (10) have studied the surfaces of the metal electrodes and waxes used in electret preparation in reflected light by Babinet's compensator and concluded that the orientation of the surface molecules depended on the cooling conditions. The generally accepted theory of the electret effect (11) explains the heterocharges as due to dipole orientation in the waxes which contain electric dipoles. The dipole orientation increases the field at the dielectric electrode interphase to such a high value as to cause breakdown at the interphase. Homocharges result thereby due to the exchange of ions or electrons between the electrode and dielectric.

From literature it was found that Shellac wax has not been used to study the electret phenomena. The results of previous chapter indicate that Shellac wax can give electrets. Hence it was decided to prepare electrets from Shellac wax and study their electrical as well as structural properties.
1. **Apparatus**

Two types of apparatuses were designed and constructed by the author for the preparation of electrets for the present study. These two types called henceforth as Type-A and Type-B are described later on in this chapter. The following requirements are satisfied with these apparatuses:
- In the Type-A apparatus, the solidification took place with the electrodes immersed in the material under study. In the Type-B apparatus the solidification took place in air. Electrets could be prepared at different temperatures with both the types of apparatuses by keeping them in an oven maintained at desired temperatures.

According to Good and Stranathan (7) electrets prepared by cooling in air give rise to clear orientation. With Type-A apparatus which was designed first was found to have certain defects. In this apparatus difficulty was encountered in removing the wax with the electrode assembly from the beaker without causing distortion. The method of heating the beaker for 2 to 3 minutes in order to detach wax is open to criticism as re-heating damages an electret to some extent. Secondly, in cutting out suitable specimens for X-ray work strains and distortions may be introduced. This difficulty was all the more important when cutting suitable specimen for taking X-ray photographs in which the field direction is perpendicular to the beam direction. In addition some difficulties were experienced while measuring the charge of the electrets also.

To overcome all these difficulties electrets were
mostly prepared by Type-B apparatus already described in chapter II.

Apparatus Type-A is an improved form of the apparatus used previously by various workers in this laboratory (12, 13). This apparatus though very simple in design and easy to operate was modified as regards the construction of spacer, electrodes and clamping devices. The various views of the apparatus are shown in figs. No. 1, 2. The apparatus essentially consists of an assembly of two electrodes (2, fig. 1) and for the preparation of electret (1 fig. 1) space in between is left. Two electrodes of exactly equal dimensions 3 x 3 cms were cut from a copper sheet of 2 mm thickness. The electrodes were scraped and finished with the finest grade of sand paper and then with a blade. Any irregularity or unevenness in the surface of electrodes would lead to errors in surface charge measurement and hence care was taken to see that the surfaces were perfectly plane and smooth.

The electrodes could be covered with any suitable metal foil when required (13 fig. 1). For this purpose, the foil was first straightened by pressing between the plane glass slabs and then firmly pressed on to the electrode. The edges of the foil were turned outwards and over the other side of the copper plates and firmly fixed there so that there remained no air gap likely to affect the preparation of electret and hence the charge measurement.

These electrodes are connected to copper rods (3 fig. 1) of 0.3 mm diameter and of length 8 cms. The connecting ends of these rods were bent at 90° and flattened so that they could
be soldered to connecting wires. The bending of rods increased the distance between them to 3 cms thus avoiding the possibility of an electrical discharge on applying high potentials through the terminals (10 fig. 1). The separation of rods to 3 cm distance was further ensured by passing them through a circular ebonite rod of 1.2 cm diameter (4 fig. 1). The rods were held tight by screws (5 fig. 1). The rods also passed through an ebonite lid of nearly 0.4 cm thickness (6 fig. 1) which covered the pyrex beaker (7 fig. 1) containing wax. These rods were further made firm by pressing them with small rectangular ebonite pieces (not shown in fig.). The ebonite cover had another hole for thermometer to pass through as shown in 11 fig. 1. The actual electret preparation was done in the slot of the Spacer (1 fig. 1) which is made of mica. A 3 x 3 x 0.1 cm cavity is obtained by the insertion of mica spacer of 0.1 cm thickness in between the two electrodes. The spacer is shown in fig 2a. When the electrode system was immersed in the molten wax, the cavity got filled with the liquid wax. The electric field was then applied for 3 hours and then the wax was allowed to solidify for 3 hours to form electret. In order to prepare electret of different thicknesses a spacer of proper thickness was introduced.

An assembly of four U shaped ebonite clips (9 fig. 1) kept the electrodes and spacer tight and bound and molten wax could not enter in between the electrode and spacer. The whole weight of the electrode system was supported by an
FIG. 1.

1. MICA SPACER  8. EBIKITE SUPPORT
2. ELECTRODES  9. EBIKITE CLIPS
3. COPPER RODS  10. TERMINALS
4. EBIKITE ROD  11. THERMOMETER
5. SCREW       12. EBIKITE INSULATION
6. EBIKITE LID  13. TIN FOIL
7. FIREX BEAKER 14. ELECTRET HOLDER
ebonite support (8 fig. 1). A suitable thermometer was inserted through a hole prepared in the ebonite lid (11 fig. (1)) for measuring the temperature of the wax inside the beaker. To ensure proper insulation ebonite was used as shown (12 fig. 1).

2. Method of Electret preparation

Preparation with Type-A apparatus

The following method was used for the preparation of electrets. The melting point of purified Shellac wax used was 77.5°C as found out by the author in the laboratory earlier. The copper electrodes were washed in hot caustic soda solution to remove adhering impurities and then with cold water. After drying, cotton dipped in benzene was used to clean the surfaces further. The surface of the tin foil also was cleaned with benzene to remove any impurity and two pieces of area 3.4 x 3.4 cm were cut out of this foil. This foil was pressed against the electrode and the excess area was bent on the other side of the electrodes and fixed there with an adhesive. The foil was well pressed against the electrode to avoid air gaps.

The spacer was introduced between the electrodes with the open end upwards. The whole system along with the lid was kept on a pyrex beaker containing Shellac wax of sufficient quantity to immerse the whole electrode system completely. While the electret was being prepared the temperature was controlled by a thermostat which kept the desired temperature constant within ± 0.5°C.
Fig. 2

APPARATUS TYPE-A
(Side view)
The thermostat was set at the desired temperature. The electrode system was removed along with the lid from the beaker and was kept inside the thermostat with all the electrical connections made through the window provided at the top of thermostat. The beaker was kept over an electric heater and when the wax melted and was thoroughly liquified at a temperature 4 or 5 degrees above the melting point of wax, this was kept in the thermostat. The electrode system already kept in the thermostat heated to the same temperature was at once immersed in this beaker. As the present investigations are mainly concerned with the preparation of electrets at different temperatures varying from 40°C (room temperature) to 90°C, the thermostat was accordingly adjusted for each electret preparation at the desired temperature.

The high D.C. voltage of desired strength i.e. 10 KV/Cm was applied by a 5 KV Ionisation tester Type -732 manufactured by Airmec Laboratories Ltd. Cressex, Bucks. The voltage is continuously variable from 0 to 5 KV D.C. It operates from 200–250 volts 50 C/S, A.C. mains. The high direct voltage is produced by the R.F.type power pack and the order of output current is 5 microamps. After 3 hours the thermostat was switched off and the temperature decreased to 40°C in three hours while the field was on. The total time for the application of field was thus 6 hours in all. Finally when the temperature was 40°C, the field was switched off and electrodes were short-circuited. After this the electret was removed from the electrodes. This was done by removing the whole solidified wax from the beaker by
Ionisation Tester & Thermostat.
slight heating. The electrode assembly was then taken out from the solidified wax block. To remove the electrodes the ebonite disc and ebonite rod were moved carefully. The electret was immediately wrapped in a tinfoil and left as such till its charge was to be measured or X-ray diffraction studies were to be undertaken. As this method was attended with difficulties as stated earlier, the Type-B apparatus was constructed.

**Type-B Apparatus**

This apparatus designed after rectifying all the defects of the type A is shown in fig. I chapter II with which most of the work was done. The apparatus described fully in chapter II, has the addition here, that positive terminal of high voltage is connected to the upper-electrode nut No.6 fig. I-a (chapter II) while the negative terminal to the lower copper plate by the nut No.3 fig.I-a.

**Preparation of Electrets with Type-B Apparatus**

This apparatus though very simple and easy to handle proved very efficient in the preparation of electret. Practically all the electrets used for measurements were prepared with the help of this apparatus.

The setting up of the electrode assembly had to be done with great care, as there was a risk of short-circuiting of electrodes when thin electrets were to be prepared. In the beginning efforts were directed towards preparing smooth and uniform samples. These minor details of preparation have a greater effect upon the subsequent properties of electret. The lower electrode was first polished by
scraping with a blade and then cleaning with benzene. The tin foil was cut exactly of the same shape and size as that of the metallic part of the lower electrodes with holes and a rectangular piece cut out. The tin foil was made perfectly plane as was done previously, and pressed to the electret surface so that it stuck there.

The lower electrode, the spacer mica sheet in which the electret was to be prepared, ebonite sheet and steel clip were bound together with six screws. Before using the upper electrode its surface and four sides were smoothed with a blade and then cleaned with benzene. A tin foil which was planed perfectly and cleaned was cut out so that its area was slightly greater than the electrode area. This foil was cut out at the four corners so that they could be folded towards the back side of the electrode and pressed to it. The wax in a small pyrex glass beaker was placed on a heater with an asbestos sheet underneath so that it may not directly come into contact with the metallic surface of the heater. When the temperature reached the melting point of wax, the electret holder was placed over the heater with the upper electrode kept separately on the heater. The time difference between keeping the wax and the electret assembly on the heater was so adjusted that both got heated up to a temperature slightly above the melting point of wax. The sides of the beaker were cleaned so that while pouring the liquid wax from the beaker, there was no obstruction which may lead to solidification of wax inside the beaker itself. It was also seen that before pouring the wax into the electret cavity, the tin foil of lower electrode had
to the upper electret surface was carefully peeled off.
By a slight pressure the mica sheet could be removed from
the lower electrode along with tin foil. The electret in
the casket was short-circuited by wrapping it in a tin foil
strip. The preservation of electret is described later in
this chapter.

**Accessories**

Kojima and Kato (17) measured the charge with-
out touching the surface of an electret. They employed a
generating voltmeter. Feaster, Prosser and Wiseman (18)
used an apparatus i.e. automatic electronic recording poten-
tiometer for measuring the charge of the electret. The
apparatus used by the author is shown in fig. 3 and 4.
It consisted of a fixed electrode which was earthed and on
which electret was placed. The upper electrode was connec-
ted to a moving system. It was made to touch the surface of
the electret and then raised to a constant height for the
measurement of charge by induction. The frame of this appa-
ratus was made of brass hollow cylinder with wall thick-
ness of 4 mm and length 16 cms (17 fig.3) The upper and
lower sides of the cylinder were closed by brass plane sheets
4 mm thick. The cylinder had an outer diameter of about
10 cm and from the lower part of it a portion of area 6 x 7
cm was cut out for a window for the insertion of electret
(18 fig.3) The lower brass circular electrode (13 fig 3)
had a diameter of 5 cm and thickness of 4 mm and it was
fitted to the bottom of the apparatus at its centre by ri-
vets. A two spring device was used for lifting the elect-
-rode. These two springs were kept apart separated by a collared brass cylinder (4 fig.3). The collared lid of the cylinder had a hole at its centre with diameter slightly greater than 5 mm so that the brass rod (7 fig.3) which was of exactly 5 mm diameter could easily move inside this. The length of this rod was 16 cm. An ebonite solid cylinder of diameter 1.75 cm and length 2.5 cm (8 fig.3) was connected to the lower side of 16 cm rod. To the other end of this rod, the ebonite holder was fitted in and the collar on the brass rod acted as a base for this holder.

The moving upper brass electrode (14 fig.4,3) had a surface area of 0.5 sq. cm and had a stem nearly 1.2 cm long. On one end of the stem threads were cut and it was screwed 0.4 mm inside the ebonite cylinder (8 fig.3) which insulated the electrode from the rest of the system. Immediately below the ebonite handle there was a bracket made of copper strip of width 0.8 mm and length 12 cm. This was bent four times to form the shape as shown in (2 fig.3). Thus the bracket had a final length of 4 cm and breadth 2 cm and remaining 2 cm were bent below on either side 1 cm each spreading on opposite sides. These bends enabled the bracket to be arrested by the two clamps (3 fig.3) fitted on the upper lid of the cylinder.

A ring was inserted to keep the lower spring away from the ebonite block (6 fig.3). This ring was clamped by a screw fitted on the brass rod. In order to adjust the pressure of the electrode on the electret, the ring was pushed either up or down and the spring (5 fig.3) was automatically pressed. To transfer the charge of electret
to the Lindeman electrometer, a terminal was fitted to the body of the apparatus (11, 12 fig. 3). This terminal was electrically isolated from the rest of the system by the ebonite screw (10 fig. 3) which was screwed to the brass cylinder. Inside this was the terminal (12 fig. 3) screwed in. Ends of the connecting wire were soldered to the electrode and to the terminal. In order to arrest the motion of the wire it was fixed to the ebonite block by a rubber band which fitted in the specially made groove on the ebonite cylinder. A terminal (15 fig. 3) was provided to earth the lower electrode and the apparatus. In Fig. 4 is the upper spring as shown, whereas (9 fig. 4) is the rubber ring.

Measurement of Surface Charge

The electret after removing the short circuit was kept over the lower electrode which was permanently connected to earth through terminal (15 fig. 3). The terminal of the measuring electrode was connected to a key system where it could be connected either to the earth or to the electrometer as desired. In order to measure the charge of electret, the mica sheet containing the electret was placed inside through the window and the bracket was pressed down and was arrested by the clamps (3 fig. 4). This made the spring ineffective due to compression (16 fig. 4). Hence the lower weaker spring (5 fig. 3) expanded and pressed the electrode down to the electret surface. In this position the upper electrode was also earthed immediately, and kept for 1 minute which meant short-circuiting of the electret. After this, immediately the bracket was released and the electrode terminal was connected to the electrometer so that the charge on the
Charge-measuring apparatus with dissicator for the preservation of electrets
electrode was shared with the electrometer. The deflection of the electrometer needle was noted with the help of a microscope. Detailed account of this is given in the next chapter.

**Preservation of Electrets**

The atmospheric conditions and seasonal changes play an important role in affecting the surface charge of electrets. Hence careful attention is necessary for preserving electrets. When they are exposed to humid air there is a decrease in charge, though the surface charge can be regained by keeping it in a dry chamber. Very prolonged exposure to humid air produces permanent damage.

Dry weather is best suited for the preparation, measurement and preservation of electrets. For preservation they were kept in a desiccator. Another important precaution is about the short-circuiting of the electrets. The charge of electret decays soon if it is kept without short-circuiting for a considerable time (6) (15). Gemant reports that measurements must be made immediately after the circuit short/have been removed as the charge decays to a considerable extent very soon afterwards. It recovers, of course, again on reapplication of the short-circuit.

The method of short-circuiting of electret is shown in fig. 5 a b c d. The electret prepared in the mica cavity was wrapped in a plane tin foil strip 15 x 2.5 sq. cm. The tin foil should remain in contact with the electret and in order to achieve this something smooth and soft had to be kept on either side of the mica sheet, so that the tin
Preservation of Electrets

Fig: - 5
foil remained pressed to the electret surface. For this a sheet of ebonite of thickness 2 mm and of the area same as that of the mica sheet was used. A thin layer of cotton was pasted over this as shown in fig. 5-b. To ensure uniform pressing on either side of the electret, a small weight was kept on the upper presser and kept in a desiccator as shown in fig.

Storage of electrets in typical ways were recently reported by Perlman (16) in 1960 who reported the effect of different shieldings on charge decay.
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