Dialysis of shellac solution

P K Banerjee*, B C Srivastava & Shravan Kumar
Chemistry Division, Indian Lac Research Institute, Namkum, Ranchi 834 010, India
Received 26 May 1994; accepted 6 October 1994

Studies on dialysis of aqueous shellac solution have been carried out for ascertaining selectivity between normal and dewaxed shellac and determining the optimum dialysis time for formation of shellac hydrosol. The escape behaviour shows that half-escape time for both the shellac is same and they may be used for hydrosol preparation. Optimum dialysis time has been ascertained by computation of time with resin concentration, pH and volume of the diffusate.

Shellac hydrosols can be prepared using cellophane membrane by adopting dialysis technique. The membrane hydrolysis of aqueous ammoniacal solution of shellac take place during formation of hydrosol. Palit ascertained the optimum time of formation of hydrosol by the qualitative method. The present study deals with the comparative behaviour of dewaxed shellac and normal shellac during dialysis and finding a suitable and simple way for ascertaining the optimum time for the formation of hydrosol, which can be used in encapsulation, a rapidly expanding technology.

Experimental Procedure

The dewaxed machine made shellac (DL grade prepared by solvent process) and normal handmade bhatta shellac (prepared by bhatta process) were used in the present study. Cellophane membrane (cellophane paper sac) was used for the dialysis of shellac solutions.

Aqueous ammoniacal solution (0.5N) of shellac (10% w/v) was prepared by dissolving shellac in warm ammoniacal water. This solution (250 mL) was taken in a suitable cellophane bag and dialysed against distilled water (250 mL), kept outside in a beaker in the controlled temperature (30°C) chamber.

For weight analysis, the concentration of resin was determined gravimetrically by evaporating to dryness a known volume (10 mL) of representative aliquot from diffusate and then heating for 2 h at 100 ± 5°C until the weight became constant. The concentration of resin in retenate was calculated after determining the resin passed out in diffusate.

The pH was determined with a pH meter (Philips model PR-9405M) by using glass and reference (saturated calomel) electrodes. The volume of the diffusate was determined with the aid of graduated cylinders. The properties 1 to 7 (Table 1) of dewaxed lemon (DL) and bhatta shellac solutions were determined by adopting the reported methods.

The infrared spectra of DL shellac, bhatta shellac and their corresponding dialysed shellac resins were obtained in KBr using Perkin Elmer IR instrument.

Results and Discussions

To observe the escape behaviour of bhatta and DL shellac, the log of the decrease in concentration of the retentate (log of % remaining) has been plotted against time (Fig. 1).

<table>
<thead>
<tr>
<th>Table 1—Characteristics of shellac solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sl. No</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>1</td>
</tr>
<tr>
<td>2</td>
</tr>
<tr>
<td>3</td>
</tr>
<tr>
<td>4</td>
</tr>
<tr>
<td>5</td>
</tr>
<tr>
<td>6</td>
</tr>
<tr>
<td>7</td>
</tr>
<tr>
<td>8</td>
</tr>
<tr>
<td>9</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td></td>
</tr>
</tbody>
</table>

*Author to whom correspondence should be addressed

Present address—P N Bose Compound, Ranchi 824 001, India
In order to compute the critical dialysis time for the formation of shellac hydrosol the resin concentration, the volume and pH of diffusate have been plotted against time (Figs 2-4).

The escape patterns, i.e., log plots (Fig. 1) of bhatta shellac and DL shellac results almost a straight line after third day of dialysis. This shows that the solute (shellac resin) behaviour is almost ideal after third day of dialysis. This finding with shellac is in agreement with the findings in polypeptides\textsuperscript{12,13}.

Further, apparent break with curvature in the escape pattern (Fig. 1) of shellac indicates involvement of another molecular species in dissociation. During dialysis most of the soft fraction of the shellac resin passes out which has also been analytically observed in the study reported earlier\textsuperscript{3} and thus corroborates the observations made in Fig. 1. A similar dissociation behaviour has also been previously reported in tetrameric haemoglobin polymer\textsuperscript{14}.

Thus, in shellac it may be said that one break in escape curve is due to the presence of a corresponding
soft resin component which rapidly dialyses out as observed in the study of dialysed shellac.

The dialysis plots (Figs 2-4) show that at first a rapid dialysing solute which is a soft fraction of shellac resin (molecular weight 550) passes and thereafter it is a bit slow. After third day of dialysis one major break has been observed in plots. It shows that two major components are present in shellac, which has already been established. Further, these plots also show that solute is homogeneous with respect to size after third day of dialysis.

The half escape time is almost the same being 4.5 days for both types of shellac (Table 1). This observation exclusively reveals that the size of the solutes is same and there is no difference in both types of shellac. It is supported by earlier IR spectral study (Figs 5 and 6). Therefore, either of these two types of shellac may be chosen for the hydrosol preparation. From the cost point of view, ordinary bhatta shellac may be preferred to DL shellac for preparing hydrosol for encapsulation processes.

The weight of the residue in diffusate has been plotted against time for computing optimum dialysis time (Fig. 2). After third day of dialysis, an abrupt shift in plots of both types of shellac has been observed. Escape curves (Fig. 1) are of similar nature to these plots. This abrupt shift corresponds to the optimum time for the formation of hydrosol which has also been ascertained by following the earlier reported qualitative method. The dialysate obtained after third day of dialysis gave slight colouration (no precipitate) with Nessler’s solution and only a faint opalescence has been observed on addition of dilute hydrochloric acid or potash alum solution. This qualitative procedure for finding optimum time of formation of shellac hydrosol was followed earlier. It has been found that the evolved quantitative and reported qualitative procedures are in close agreement with each other.

The volume of the diffusate has been plotted against time for computing optimum dialysis time. An abrupt shift in plots (Fig. 3) has been observed after third day of dialysis in both types of shellac. This criterion may be used for ascertaining optimum time of formation of good sol as it is also in agreement with the above as well as reported qualitative methods.

The pH plays an important role in ascertaining the optimum time for the formation of a good sol. Therefore, the pH of the diffusate has been plotted against time. The pH remains fairly same (pH 7, approx.) after third day of dialysis. A shift in the curves (Fig. 4) of both type of shellac has been observed after third day of dialysis. Therefore, it may also serve as a suitable criterion for formation of a good sol. Moreover, it also coincides with the observations reported in above paragraphs. The summarised characteristics of both type of shellac solutions are presented in Table 1.

It has also been observed in these dialysis experiments that at the beginning of dialysis-run when the concentration of solute is highest inside the bag (sac), water tends to flow into the sac due to osmotic pressure and induces dilution of the hydrosol which becomes progressively less as solute moves out. The effect of dilution of the hydrosol is apparent in
escape plots (Figs 1 and 2) indicating a decrease in solute concentration against time. The increase in volume inside the sac is usually not more than 40%. These are also the reasons for getting linear escape rate. Similar observations have been reported for protein macromolecules.

Conclusion

Dialysis studies suggest that either normal bhatta shellac or dewaxed lemon grade shellac may be used for shellac hydrosol preparation. It has been found that the volume or resin concentration or pH of the diffusate may be used as a suitable criterion for finding the optimum dialysis time for formation of shellac hydrosol. The evolved quantitative methods are precise and are in close agreement with earlier reported qualitative method.

Acknowledgement

Authors are thankful to late Prof. R P Kapil, Dr T P S Teotia, exdirector and Dr B B Khanna for their keen interest in the work. The help rendered by Dr A Pandey, Senior Scientist in determining the specific conductivity of shellac solutions is also duly acknowledged.

References

1 Palit S R, J Ind Chem Soc, 17 (1940) 375.
2 Palit S R, Indian Lac Res Inst Bull, 40 (1940) 1-5.