# Selection of an Optimal Surface for the Preparation of Uniform Detached Films of Adhesive Materials

*Sir:*

Shellac films are used extensively in the pharmaceutical industry in sugar-coated tablets as waterproof barriers, in enteric medication, and in sustained-release dosage forms. These polymeric films often vary with time because of complex chemical and physical changes, which may be the cause of erratic drug-release patterns observed in several dosage forms.

Studies of the aging of shellac films can be handicapped seriously when they are restricted to applied films, since this procedure can introduce exogenous variables which may lead to erroneous or misleading results. In addition, many properties of the film itself cannot be evaluated after application.

The development of a detached shellac film presents a particularly difficult problem. Shellac displays a high degree of adhesion to surfaces used routinely for film casting. Removal of the cast and dried film from these surfaces can result in destruction of film integrity.

Surfaces such as glass, stainless steel plates, chromium-plated steel plates, celluloid, and formica were evaluated in our laboratories and rejected as inadequate to overcome this problem. Attempts to utilize Gardner's (1) technique of casting on a mercury surface met with only moderate success. Wax-free shellac glazes could be spread on a mercury surface, but a small amount of mercury adhered to the detached dry film. In addition, film thickness was extremely difficult to control. On the other hand, glazes containing natural wax possess a sufficiently high interfacial tension against mercury, so that a negative spreading coefficient results, and they could not be spread uniformly on a mercury surface.

To prepare films of uniform thickness from most types of shellac, a surface upon which the shellac glaze demonstrates a positive spreading coefficient and a low work of adhesion was required. Therefore, the decision was made to investigate silicone-treated paper as a possible film casting surface. These papers are available in a wide range of silicone content and antiadhesive quality. A series of 8 × 11 in. silicone-treated papers was mounted on a level formica-plated platform. Solutions containing 40% (w/w) shellac resin,[1] suitably plasticized with 2 to 5% (w/w) dibutyl phthalate in anhydrous ethyl alcohol, were freshly prepared and cast on the treated papers according to the method described by Kanig and Goodman (2).

The solutions used in this study were prepared from samples of the two basic types of shellac resins: one type is produced by a chemical bleaching process, and the other is purified by mechanical means. Each type was tested with and without the natural wax content. After casting, the films were dried under ambient conditions or in ovens at various temperatures for a period of 24 hours, then manually removed.

Optimum results were obtained utilizing Riegelease MF separator paper No. 53044 for both types of wax-free shellac and Riegelease paper No. J-4043 for both types of wax shellac.[2] The latter paper represents a more highly treated silicone surface. In each case, neither the casting of the glaze nor the removal of the dried film posed difficulty. Test films of 5 and 10 mil wet thickness were readily cast and easily detached. Silicone adherence to the dried films was not evidenced. A high degree of uniformity of the dry film was observed. The dry film thickness, measured by means of an Ames model 2W dial comparator to an accuracy of $10^{-4}$ in., varied less than ±10%.

Providing that caution is exercised to insure that the solvent system employed has no affinity for the silicone resin impregnated in the paper, the silicone-treated surfaces appear to offer a considerable advantage because, with a minimum of procedural screening, an optimal surface for the casting of free films may be selected quickly. It is suggested that this approach will greatly simplify the study of highly adhesive, tenacious, film-forming polymers.

(1) Gardner, W., and Kappenberg, W., *Ind. Eng. Chem.*, 28, 437(1936).
(2) Kanig, J. L., and Goodman, H., THIS JOURNAL, 51, 77(1962).

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