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STABILITY OF THE VISCOSE TYPE OF OZAPHANE PHOTOGRAPHIC FILM

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ABSTRACT

Viscose Ozaphane, a new type of film with a base of regenerated cellulose sheeting, and having certain advantages for record use, was tested to determine its comparative stability. Its stability was compared with that of cellulose nitrate, and also with that of cellulose acetate, which is widely used for film-slides, and which has been found to be a very stable material for preserving records in libraries. The viscose type of film apparently is not suitable for permanent records, but does appear to have properties to recommend its use for reading-room copies that can be replaced when they become unserviceable. The stability was determined by measuring changes in its chemical and physical properties under accelerated aging. The changes observed were increase in acidity and copper number, and decrease in viscosity, weight, and flexibility.

CONTENTS

I. INTRODUCTION

Viscose Ozaphane, a relatively new type of motion-picture film, was investigated with reference to its suitability for use as a record material. In this new type of film, a light-sensitive dye incorporated within the base performs the function of the emulsion coating on the conventional type of film now in use. The film tested had a base of transparent viscose sheeting about 0.002 inch in thickness, plasticized with glycerin to obtain greater flexibility. The new type of film is of particular interest for record purposes for several reasons. It is grainless, gives high contrast, is slow-burning, the image is not readily damaged by scratching, and the film is only one-third as thick as the films of the emulsion type. The cellulose-acetate film now used for

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record purposes has been found in previous studies to be [2] a very stable material when properly made [3] and properly processed photographically. The purpose of this study was to determine the relative stability of this type of Ozaphane film.

II. DETERMINATION OF STABILITY BY ACCELERATED AGING

The stability of the new type of film was determined by measuring changes in its chemical and physical properties under accelerated aging. The accelerated-aging treatment used was the same as that previously employed in evaluating acetate and nitrate films, namely, that of heating in dry air at 100° C. This test had formerly been found suitable for paper.[4,5] Loss of flexibility of papers and films, measured by a folding-endurance test, is the most significant property, because the flexibility is most sensitive to changes produced by deterioration. Measurements of the loss in weight on heating were also made. Other tests consisted in determinations of the decrease in viscosity of solutions of the film, and increase in the copper number and in acidity measured as pH. To assist in interpretation of the results, data previously [6] obtained for acetate and nitrate films under comparable aging treatments are included in the graphs for purposes of comparison.

III. EFFECTS OF ACCELERATED AGING

1. LOSS OF FOLDING ENDURANCE

The Schopper folding-endurance tester, which is an instrument widely used for determining the folding endurance of paper, was used to measure the effects of aging on the flexibility of the film. Results of tests with the MIT folder indicated that it was unsatisfactory because of excessive stretch of the specimens under test; and the film was found to be too thin to be successfully tested in the Pfund tester which had been used for acetate and nitrate films. All folding tests on the Ozaphane film were made under constant atmospheric conditions of 50-percent relative humidity and 75° F, the conditioning time being 24 hours for all specimens. The standard humidity of 65 percent was not selected, because stretch of the specimens under test was troublesome at that humidity. Control and aged specimens were conditioned and tested under identical conditions, and all specimens were humidified by adsorption to avoid variations attributable to hysteresis in moisture sorption.

The effects of oven aging on the folding endurance of Ozaphane as compared to acetate- and nitrate-base films are shown graphically in figure 1. The results show a more rapid loss of flexibility under the heat test for Ozaphane than for the acetate-base film. It was, however, much more resistant than the nitrate-base film. After 30 days of oven aging, the acetate film retained approximately 67 percent of the original strength, while Ozaphane retained only 13 percent. None of the nitrate film retained any measurable folding endurance after 15 days of heating.

[2] J. Research NBS **17**, 871 (1936) RP950.
[3] Misc. Pub. BS M158 (1937).
[4] BS J. Research **3**, 476 (1929) RP107.
[5] Paper Trade J. **95**, 28 (July 1932).
[6] J. Research NBS **17**, 871 (1936) RP950.

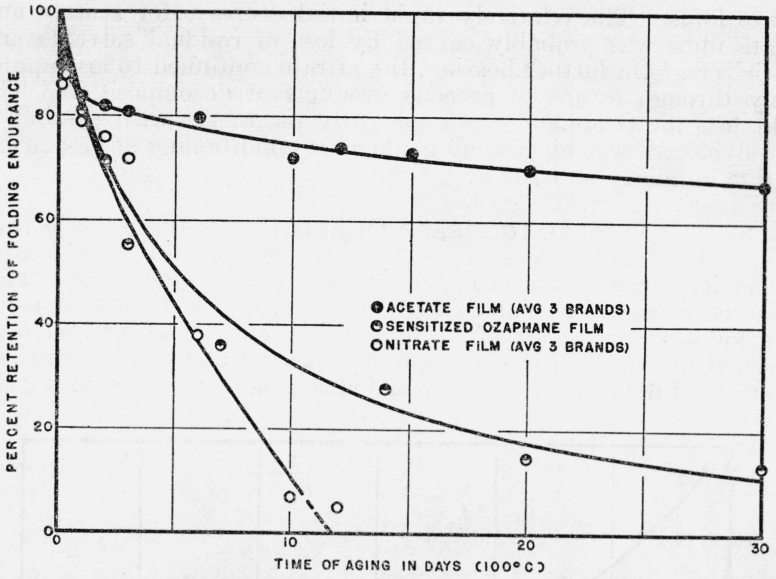

FIGURE 1.—*Effect of oven aging on flexibility of viscose Ozaphane film as compared to effects of similar treatment on acetate and nitrate film.*

2. LOSS OF WEIGHT

Oven aging caused losses in weight for all types of films. However, the Ozaphane type showed only slight losses even for extended periods of heating. Figure 2 shows loss in weight for Ozaphane, acetate, and

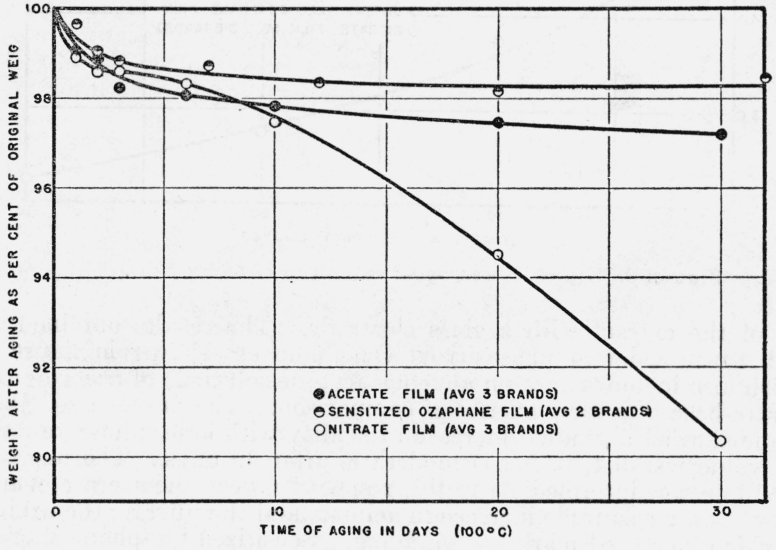

FIGURE 2.—*Loss in weight during oven-aging of viscose Ozaphane, acetate, and nitrate films.*

nitrate films. The relatively rapid initial decrease for acetate and nitrate films was probably caused by loss of residual solvents and plasticizers. On further heating, the nitrate continued to lose appreciably through escape of gaseous products of decomposition. The slight loss for Ozaphane was apparently plasticizer with possibly a little dye. All weighings were made after conditioning at 65-percent relative humidity and 70° F.

3. INCREASE IN ACIDITY

Chemical degradation of cellulosic materials is often accompanied by increased acidity. The changes in acidity of Ozaphane with aging were followed by means of pH determinations. The method used consisted in heating 1 g of the ground material in 100 ml of distilled water of pH 6.0 to 7.0 for 1 hour in a steam bath, then measuring the

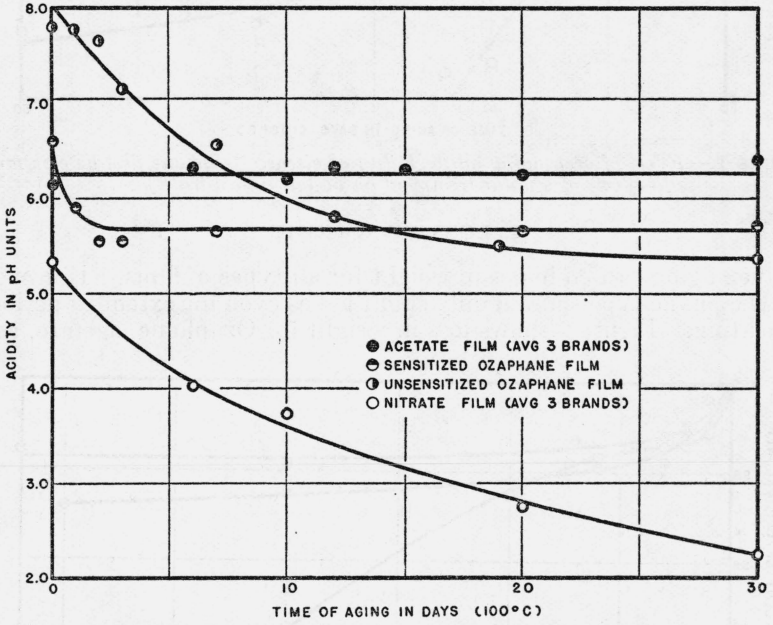

FIGURE 3.—*Effect of oven aging on acidity of record films.*

pH of the extract with a glass electrode. The results obtained on both sensitized and unsensitized Ozaphane are shown in figure 3, which also includes data on aqueous acetone solutions of acetate- and nitrate-base film for purposes of comparison. The unsensitized Ozaphane showed a gradual increase in acidity with aging; however, the pH value was 5.4, which is moderate, after 30 days. The unsensitized film was intermediate in this respect between the acetate, which showed no measurable increase in acidity, and the nitrate, the acidity of which increased markedly on aging. Sensitized Ozaphane showed little change of pH after 1 day of heating, possibly because of buffering action of the dye in the film, some of which went into solution during extraction.

4. INCREASE IN COPPER NUMBER

The copper number of cellulose is defined as the number of grams of copper reduced from the cupric to the cuprous state by 100 g of the material under defined conditions. An increase of copper number is accepted as indicative of degradation of a cellulosic material; hence, the rate of increase during accelerated aging is considered a measure of stability. Figure 4 shows the effects of accelerated aging on the copper number of unsensitized Ozaphane film and acetate-base film. The tests were made in accordance with the official method of the Technical Association of the Pulp and Paper Industry.[7] Unsensitized Ozaphane was used for the copper-number determinations to avoid errors introduced by the reducing effect of the dye in the sensitized film. Although the copper number for unaged acetate-base film is relatively high, it changes very slowly on aging, the increase

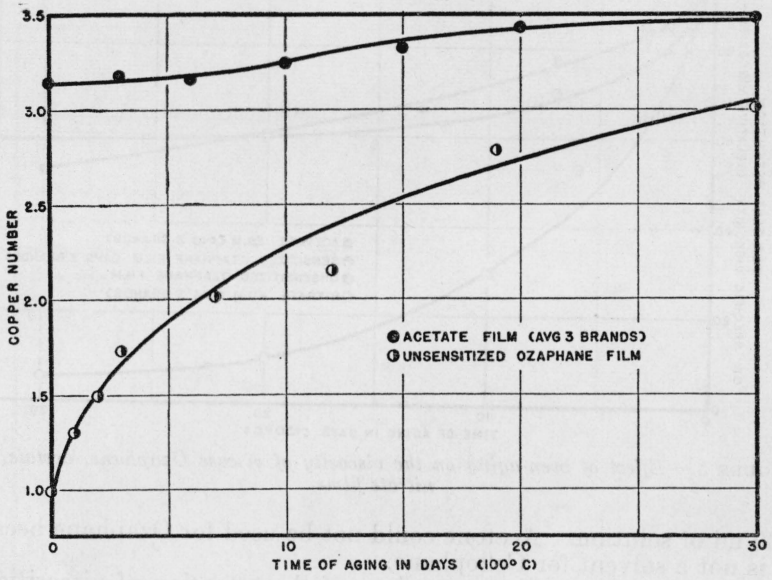

FIGURE 4.—*Change of copper number during oven aging of viscose Ozaphane, and acetate films.*

being 10 percent for 30 days' aging. The increase for Ozaphane was 200 percent for the same treatment, which indicates definitely poorer stability under oven aging.

5. DECREASE IN VISCOSITY

Data on the viscosity of solutions of cellulosic films provide the most reliable measure of their chemical degradation. According to Staudinger,[8] the specific viscosity of long-chain molecules is directly proportional to the molecular weight, for dilute solutions of equal concentration. Molecular break-down should therefore be accompanied by a proportional decrease in specific viscosity. Clibbens and Ridge [9] have shown that decreases in strength of cotton fibers

[7] TAPPI Standard T430m, Tech. Assn. of the Pulp and Paper Ind.
[8] H. Staudinger, Die Hochmolecularen Organischen Verbindungen (Berlin, 1932).
[9] J. Text. Inst. 19, 390T (1928).

produced by a variety of reagents are accompanied by corresponding decreases in the viscosities of their solutions.

The effects of accelerated aging on the viscosity of Ozaphane were determined by measuring the viscosities of solutions made before and after various aging periods. The procedure used was that recommended by the British Fabrics Research Committee [10] and described by Clibbens and Little,[11] except that the concentration of ammonia in the standard cuprammonium solvent was 240 g per liter, as recommended by Clibbens and Geake.[12] The measurements were made at $21 \pm 0.05°$ C, using solutions containing 2 g of dry Ozaphane per 100 ml of solution. Hill and Weber [13] determined the viscosities of acetate and nitrate films by using acetone solutions containing 1 g of film per

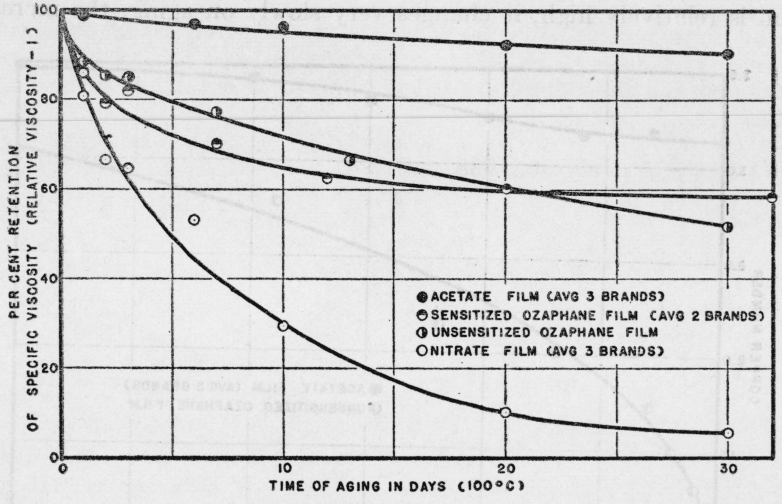

FIGURE 5.—*Effect of oven-aging on the viscosity of viscose Ozaphane, acetate, and nitrate films.*

100 ml of solution. Acetone could not be used for Ozaphane because it is not a solvent for Cellophane.

Figure 5 is a graphic comparison of the retention of viscosities of acetate, Ozaphane, and nitrate films. The acetate retained over 90 percent of its original specific viscosity after 30 days of aging, while the Ozaphane films retained less than 60 percent and the nitrate 6 percent. Here again, the Ozaphane is intermediate in chemical stability between the stable acetate-base and unstable nitrate-base films.

IV. SUMMARY AND CONCLUSIONS

The data indicate that the Ozaphane type of film having a viscose base is definitely inferior to good acetate film as regards stability under accelerated aging. It is not suitable for permanent records, but apparently it has sufficient stability for positives for reading-room use. Its stability is comparable to that of ordinary sulfite wood-fiber

[10] The Viscosity of Cellulose Solutions. Fabrics Research Comm., Dept. Sci. and Ind. Research, London (1932).
[11] J. Text. Inst. **27**, 285T (1936).
[12] J. Text. Inst. **19**, 77T (1928).
[13] J. Research NBS **17**, 871 (1936) RP950.

papers, which are known to last 25 years or longer under ordinary conditions if they are well made. Since the reading-room copies that are in use will doubtless become largely unserviceable from mechanical wear in less than 25 years, greater stability for that purpose does not appear essential. The Ozaphane type of film has certain advantages for use as positives: It is grainless, gives high contrast, is only one-third as thick as acetate, and has no emulsion to become scratched during projection and handling.

Although the viscose-base film apparently is not sufficiently stable for permanent records, it is not designed for use where the highest permanence is required. Negative films are in reality the master records, and the dyes employed at present in Ozaphane are too slow to permit its use for original photographs.

It is quite possible that the stability of the Ozaphane type of film can be improved by using for a base a sheeting having higher initial purity. It is understood that a film of this kind has recently been developed and investigation of it is planned as a further part of the work.

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