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The effect of the temperature of preparation on the mechanical properties and structure of gelatin films

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[Plate 4]

Gelatin films prepared by evaporation from aqueous solution at temperatures of 60° C and above differ considerably in structure and mechanical properties from films dried slowly at 20° C, when gelling precedes dehydration. In the high-temperature preparation dehydration precedes the formation of a continuous structure, and X-ray and other evidence indicates that the molecular chains are in a disordered contracted state not far removed from their condition in the sol. The high-temperature film is characterized by low strength and high recoverable extension under conditions of high relative humidity. In the low-temperature preparation the greater degree of crystallization has partially extended the molecular chains, and the unidirectional contraction of the film on drying has oriented them in the plane of the film. This film exhibits thermal contraction in hot methanol, and is stronger, but at high humidity is much less extensible, than the high-temperature preparation.

Of the adhesives used for sizing rayon textile yarns, gelatin is the most common, and probably more is known about its sizing and weaving behaviour than about that of other adhesives. It is thus a reasonable starting-point in a long-range

fundamental study, the ultimate aim of which is to relate the physical properties of the size film to sizing and weaving behaviour. Although the literature abounds with accounts of work on gelatin in solution and in the gel state, there are fewer accounts of work on gelatin films, and none has been found dealing specifically with the effects of the temperature of preparation on the mechanical properties of the films.

For the experiments described below, a commercial acid-processed skin gelatin was used. Its isoelectric point was at pH 6·5, and a 5 % solution had a pH of 4·2. Films about 0·1 mm thick were prepared by evaporation of water from 5 % solutions contained in shallow metal trays, and the conditions in which this evaporation took place were varied as described later. Test-pieces of approximate dimensions 5 cm × 6 mm were then cut, and conditioned in atmospheres of controlled humidity and temperature before being submitted to tensile tests. For the tensile tests a rate of loading of about 25 Kg/cm²s was used. At least ten specimens were tested from each sample, and the load-extension curves, tensile strength and extension figures given later are means of these.

Effect on the tensile properties of the temperature of preparation and of the humidity during testing

Films were prepared by drying in atmospheres differing in temperature but having the same relative humidity as follows:

(*a*) 20° C, 70 % r.h.

(*b*) 60° C, 70 % r.h.

The film (*a*) took about four days to become sufficiently dry for removal from the tray, whereas the film (*b*) reached this condition in about six hours. In the early stages of the drying of (*b*) the temperature in the drying film fell to 56° C owing to the rapid rate of evaporation. Thus a range of temperatures (56 to 60° C) was covered during the drying, and this also applies, but over a wider range, to films dried at still higher temperatures described later.

Table 1. Film preparation temperature and the ambient humidity during testing: effect on tensile properties

film preparation temperature (° C)	testing r.h. (%)	tensile strength (Kg/sq.cm) mean	tensile strength (Kg/sq.cm) s.d.	extension at break % mean	extension at break % s.d.
20	45	910	48	1·9	0·2
56 to 60	45	654	31	1·1	0·1
20	55	832	52	2·2	0·3
56 to 60	55	585	33	1·5	0·1
20	65	646	59	3·7	0·7
56 to 60	65	350	22	1·7	0·2
20	75	418	29	10·2	1·7
56 to 60	75	172	36	46·1	5·6
20	85	170	38	23·8	3·6
56 to 60	85	81	11	129	20

(s.d. = standard deviation.)

Both types of film were conditioned and subsequently tested under different humidity conditions covering a range from 45 to 85 % r.h. The mean figures for tensile strength and percentage extension at break are given in table 1, and the load-extension curves in figures 1 and 2.

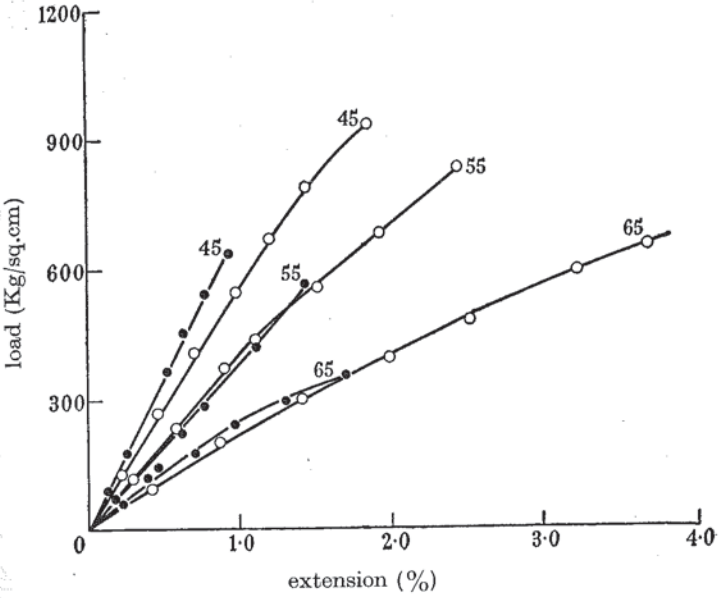

FIGURE 1. Load-extension curves at relative humidities of 45 to 65 %, indicated on the curves. ○, Film (*a*) dried at 20° C; •, film (*b*) dried at 56 to 60° C.

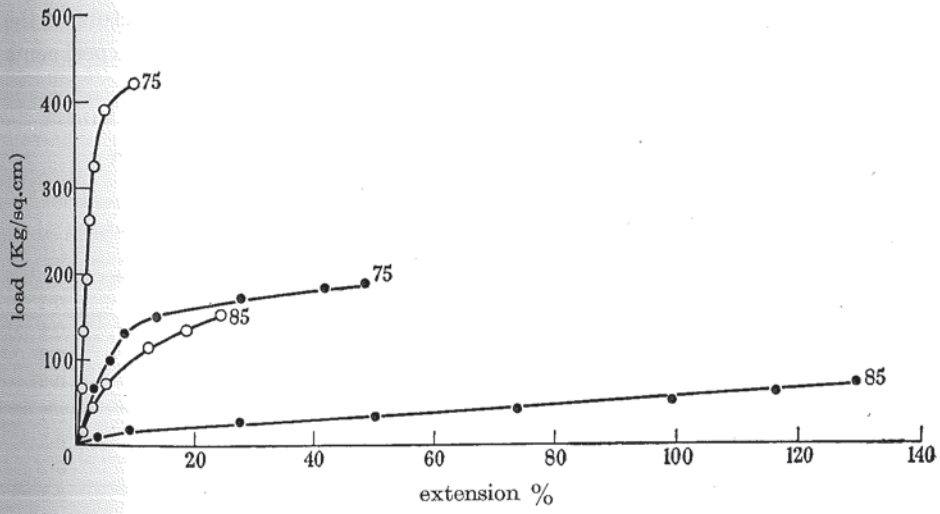

FIGURE 2. Load-extension curves at relative humidities of 75 and 85 %. ○ and • as on figure 1.

It is seen from the table that increasing the drying temperature from 20° C to about 60° C had a profound effect on the tensile properties. At all humidities the low-temperature preparation (*a*) was much stronger than the preparation (*b*), and

up to 65 % r.h. had also a greater extension at break. At 75 and 85 % r.h. the extension of the film (*b*) was much greater than that of the film (*a*).

Up to 65 % r.h. (figure 1) the load-extension curves show no obvious yield point, and the extension at break was very low for both types of film.

At 75 and 85 % r.h. (figure 2) the curves show an apparent yield point, beyond which the extension per unit load is greater than in the initial part of the curve. This extension is of a different order of magnitude from that obtained up to 65 % r.h., and increases rapidly with increasing humidity. An important point is that, even when the film (*b*) had been extended to 130 % of its length, it returned in time to within a small percentage of its original length on removal of the load. It is evident that, with the fairly high rate of loading used, there was little plastic flow during the extension of the film.

The magnitude of the effect of humidity on the tensile properties is of some significance and deserves comment. The greater effect was shown by the high-temperature preparation. For an increase in relative humidity from 45 to 85 % it suffered a fall in strength to approximately one-eighth of its initial value, while the extension at break was increased a hundred-fold.

Effects on the tensile properties of the rate and degree of drying

In the previous experiment the results show that films (*a*) and (*b*) differed considerably in mechanical properties. The cause was assumed to be the difference in preparation temperature, although related differences occurred in two other factors. These were the rate of drying, and the moisture content of the film when equilibrium was attained with the moist atmosphere, which may be called the degree of drying. The possible independent contributions of these factors to the difference in mechanical properties could not be ignored. The experiments

Table 2. The rate of drying of the film, and the ambient humidity during drying: effect on tensile properties at 65 % r.h.

factor investigated	ambient conditions during film prep. temp. (°C)	ambient conditions during film prep. %r.h.	approximate drying period	tensile strength (Kg/sq.cm) mean	tensile strength (Kg/sq.cm) s.d.	extension at break (%) mean	extension at break (%) s.d.
rate of drying	20	70	4 days	647	59	3·7	0·7
	20	40	6 h	642	42	3·6	0·4
	60	70	24 h	348	23	2·0	0·3
	60	70	6 h	350	22	1·7	0·2
ambient humidity of preparation	20	70	—	647	59	3·7	0·7
	20	40	—	650	35	3·9	0·5
	60	70	—	350	22	1·7	0·2
	60	20	—	352	19	2·1	0·3

described below were carried out to provide a rough estimate of the magnitude of the effects of these complicating factors. In each experiment the films after preparation were conditioned and tested at 65 % r.h.

The effect of rate of drying was investigated at both temperature levels (20 and 60° C) by varying either the degree of ventilation or the atmospheric humidity

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