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Comparative Rheomechanical and Adhesive Properties of Two Hydrocolloid Dressings: Dependence on the Degree of Hydration

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ABSTRACT

Hydration is believed to modify the mechanical properties (resistance to stress) and the rheological (viscoelastic) properties of hydrocolloid dressings. Rheomechanical properties in turn influence adhesional properties. In this work the relationships between the degree of hydration and the rheomechanical and the adhesive properties of two different commercial dressings (regular and nondispersive) were investigated. The two dressings showed different hydration characteristics. The regular dressing takes up water rapidly and tends to reach saturation after 12 hr. As the dressing swells and loses its integrity there is an accompanying reduction of both rheomechanical and adhesive properties. The nondispersive dressing exhibits a linear water uptake profile over extended periods of hydration up to 4 days. It does not swell and its rheomechanical and adhesive properties remain unchanged after hydration. The comparative evaluation of the results obtained with the various methods points to the relevance of mechanical and viscoelastic properties to the functionality assessment of dressings.

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INTRODUCTION

The functional properties of composite hydrocolloid dressings for skin or mucosal application are of current interest. Absorption characteristics and adhesive properties of the components are the most important parameters in terms of dressing performance. The hydration of the hydrophilic components of the dressings promotes modification of their physical structure. The increased chain mobility of the hydrophilic polymers following hydration promotes their diffusion properties and allows the establishment of adhesive interactions at the dressing–skin (or dressing–mucosa) interface; however, an excessive amount of absorbed liquid may impair dressing integrity and shorten the time that the dressing is in place.

It is conceivable that the modifications to physical structure, which are induced by hydration, cause changes in both mechanical properties (resistance to stress) andthe rheological (viscoelastic) properties of hydrocolloid dressings; these rheomechanical changes may in turn influence adhesive properties.

While many papers have dealt with in vitro tests for either moisture (1-3) or liquid uptake (4,5), little attention has been paid to the evaluation of the rheological and mechanical properties of dressings after hydration. A review of relevant literature has been published by Ferrari et al. (6).

The aim of the present work was to establish the interrelationships between the degree of hydration, and the rheomechanical and the adhesive properties of the dressings.

In this study two different commercial hydrocolloid wound dressings (Granuflex® and Granuflex® Improved) with differing hydrophilic properties were examined. Rheological properties were measured by dynamic oscillatory tests and expressed as G' (storage or elastic modulus) and G'' (loss or viscous modulus). The mechanical properties were measured by means of a gel strength tester (and expressed as resistance to compression) and with a tensometer (and expressed as resistance to elongation stresses). Adhesion potential was tested by means of a tensile stress test and expressed as work of adhesion. Measurements were carried out both on dry samples and on samples hydrated at different levels by means of a capillary water uptake apparatus (7).

The combined use of different testing methods should allow a thorough characterization of dressing functionality.

MATERIALS

The hydrocolloid dressings compared in this study were as follows:

Regular dressing (Granuflex, ConvaTec Ltd., Ickenham, Middlesex, U.K.). It consists of a thin layer of a semi-open-cell polyurethane foam bonded onto a polyurethane film, which acts as a carrier for the hydrocolloid base. The base is composed of gelatin, pectin, and sodium carboxymethylcellulose, dispersed in an adhesive polyisobutylene base.

Nondispersive dressing (Granuflex Improved, ConvaTec Ltd., Ickenham, Middlesex, U.K.). The qualitative composition of the hydrocolloid base is similar to the regular dressing, although the hydrocolloid amount is reduced. The composition and the structure of the adhesive matrix are different: in particular, a tackifying resin and a butyl rubber are present. The adhesive matrix provides a more rigid structure for the hydrocolloid gel forming phase.

METHODS

Hydration Measurements

Capillary water uptake was measured continuously over 4 days by means of a modified Enslin apparatus as described by Ferrari et al. (7). At predetermined time intervals (2, 4, 6, 16 hr for the regular dressing and 2, 4, 6, 12, 24, 48 hr, and 4 days for the nondispersive dressing) the amount of water taken up (milligrams) was measured and divided by the sample surface area exposed to water (200 mm^2) to give water uptake per unit area of dressing.

Resistance to Compression

The mechanical resistance to compression was measured by means of a gel strength tester (8). The measurements were effected on 17 mm diameter dressing sample disks (surface area 200 mm^2) both in the dry state and after hydration in the capillary water uptake apparatus (for the predetermined durations cited above).

The gel strength tester basically consists of a probe and of a microbalance which is connected to a computer. The probe is pushed into the dressing (which is placed on the balance with the absorbing side upwards) at a constant speed (4 mm/min) by means of a motor. As the probe is lowered, a force is measured by the balance and is recorded. The increase in force with time is a function of the mechanical resistance of the sample to the penetration of the probe. The gel strength parameter (newtons/millimeter) is calculated as the maximum value of the penetrating force divided by the probe displacement at this point (8).

Resistance to Elongation

Determination of the mechanical resistance to elongation was performed by means of a Tensometer 10 Monsanto (Edison Road, Dorcan, Swindon, Wiltshire SN3 5HN, U.K.).

Dressing samples, 50 mm × 10 mm, were tested in the dry state and after hydration (for the predetermined durations cited above). The samples were clamped in the jaws of the tensile testing machine and stretched at different elongation rates, i.e., 15, 25, 50, and 100 mm/min. The apparatus provides a continuous output of stress and strain: stress is calculated by dividing the applied force by the original cross-sectional area of the sample and strain is the ratio of sample elongation divided by the original length of the test section of the specimen.

The stress value (megapascals) increases during the experiment up to the breakpoint (the maximum applied stress at which the sample breaks). This expresses the mechanical resistance of the dressing to elongation.

Rheological Measurements

Dynamic rheological measurements were performed using a rotational rheometer (Bohlin CS Rheometer, Bohlin Instruments Division, Metric Group Ltd., Cirencester, U.K.) connected to a personal computer (Bull Micral, Bull, Milan, Italy) for setting analysis parameters and processing and recording data. Plate-plate combination was used as measuring system. Measurements were carried out on 20 mm diameter dressing disks (surface area 314 mm^2) both in dry state and after hydration (for the previously stated durations). All measurements were carried out at 37°C after a rest time of 3 min. Dynamic oscillatory tests were performed in the linear viscoelasticity range. The viscoelastic parameters storage or elastic modulus (*G*′) and loss or viscous modulus (G″) were measured at frequencies ranging between 0.01, 0.1, and 1 Hz.

Adhesion Measurements

The adhesion potential of the two dressings was determined by means of a tensile stress tester, designed and manufactured in the laboratory in Pavia, which is represented schematically in Fig. 1.

The apparatus is assembled in a horizontal supporting base. It consists basically of a load cell (A) (mod. 524, DS Europe, Milan), which is fixed to a carriage (B) and is connected to a personal computer (IBM AT, IBM, Milan) via an amplifier (mod. DS 567 EA, Ds Europe, Milan). The carriage can be moved on two rods, which are fixed to the supporting plane. A motor (C), equipped with a speed transformer, moves a screw, which in turn pushes on the load cell; the movement is thus transmitted to the carriage.

A LVDT transducer (D) (RS n. 646-599, RS Supplies, Corby, U.K.) is also linked to the movable carriage and is connected via an amplifier (RS Transducer Conditioner Type OD3, Corby, U.K.) to the same personal computer. The LVDT transducer was calibrated by means of a micrometer device (9) in order to quantify the relationship between the electric output (volts) and the displacement (millimeters).

At the beginning of the experiment, a 17 mm diameter dressing sample disk (E), either dry or hydrated for the predetermined time intervals described above, is glued with the backing membrane in contact to a sample holder (F), which is fixed to the supporting plane. A filter paper disk (G) (17 mm diameter) is fixed, faced to the dressing, on the movable carriage; the paper disk is hydrated with 50 μl water. The carriage is then

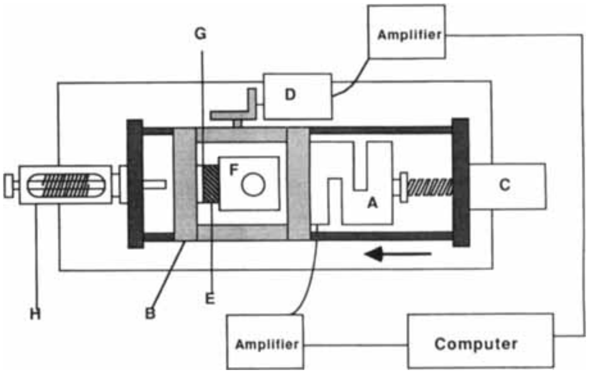

Figure 1. Schematic drawing of the tensile stress tester: A = load cell; B = movable carriage, C = motor; D = LVDT transducer; E = dressing; F = sample holder; G = paper filter; H = preload device.

moved until contact between the adhesive side of the dressing and the paper disk is established. Afterwards, a preload of 300 mN is applied to the sample by threading a special device (H) (consisting of a piston surrounded by a calibrated spring) against the movable carriage. The preload value is chosen as the minimum force which ensured an even contact between the two surfaces (paper disk and dressing).

After application for 3 min, the preload is removed and the movable carriage is moved forward at a constant speed of 4 mm/min up to the complete separation of the two surfaces. Both displacement (measured with the LVDT transducer) and force of detachment (measured with the load cell) are recorded simultaneously and logged by the computer. Force versus displacement curves were analyzed subsequently in order to obtain the maximum force of detachment and to calculate the work of adhesion from the area beneath the curve (using the trapezoidal rule).

RESULTS AND DISCUSSION

Degree of Hydration

The water uptake curves of the dressings are shown in Fig. 2. Each profile is the mean (± *SE*) of three replicates. The two samples show different hydration characteristics. Whereas the regular dressing takes up water rapidly at the start of the experiment and approaches saturation after 12 hr, the nondispersive dressing exhibits a linear water uptake profile over extended periods of hydration with no evidence of saturation even after 4 days. This shows that initially the amount of water taken up by the nondispersive dressing is less than that taken up by the regular dressing, but this situation is reversed at longer times.

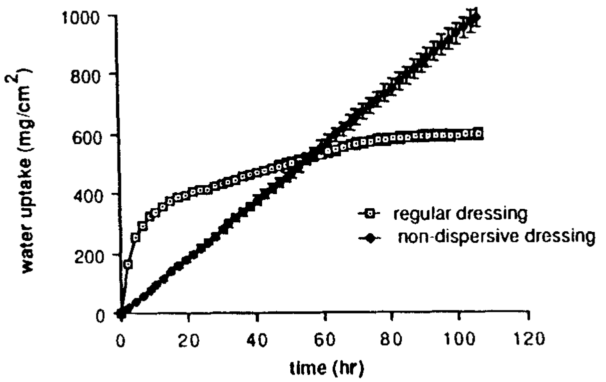

Figure 2. Water uptake profiles of the two dressings.

Both products contain the same hydrocolloids, but the improved dressings has additional polymers which produce a more rigid structure of the adhesive matrix. While the fibrous sodium carboxymethylcellulose provides a track for hydration, full hydration is prevented by the rigid structure. Thus, the matrix appears to control the rate of liquid uptake and prevent the dressing losing its integrity at high liquid levels.

Resistance to Compression

In Figs. 3(a) and 3(b) the relationship between the gel strength values and hydration level (i.e., the amount of water taken up after a certain period of hydration) are shown. Each value is the mean (± *SE*) of three replicates.

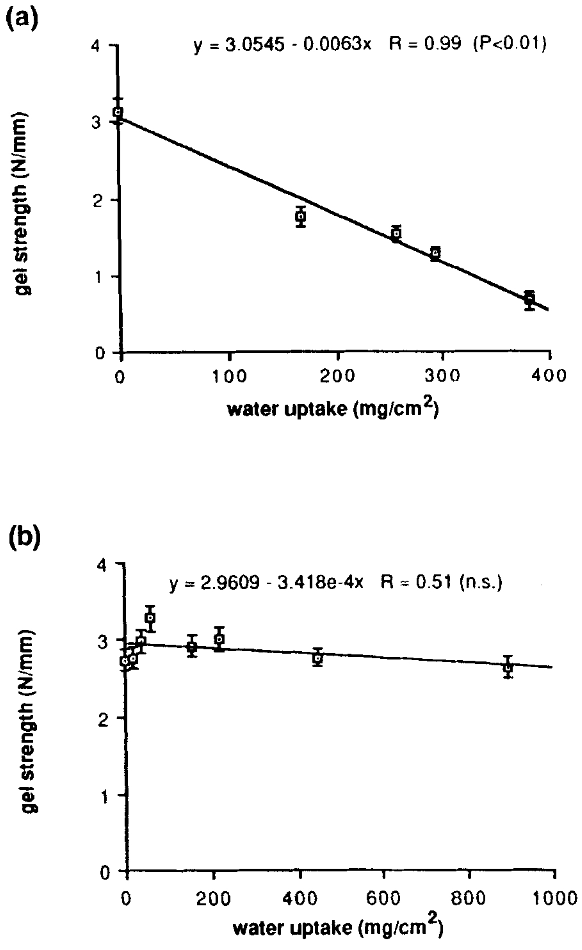

Figure 3. Relationship between the gel strength values and the hydration level: (a) regular dressing; (b) nondispersive dressing.

For the regular dressing [Fig. 3(a)] a linear decrease in gel strength is observed on increasing degree of hydration. This indicates a reduction in dressing integrity; water penetration causes the formation of a gel layer which becomes softer with increasing hydration time and therefore offers a decreasing resistance to the penetration of the probe. The loss of dressing integrity is confirmed by visual inspection of the specimen. For the nondispersive dressing [Fig. 3(b)] the gel strength remains almost constant at all hydration levels, even those reached after long time periods (more than 4 days). This can be explained assuming that the more rigid structure of this dressing prevents hydrocolloids from swelling, thus allowing the maintenance of dressing integrity after hydration.

Resistance to Elongation

Figures 4(a) and 4(b) show the influence of the rate of elongation during the test on the tensile strength of the dressings measured at increasing degree of hydration. Each value is the mean (± *SE*) of three replicates.

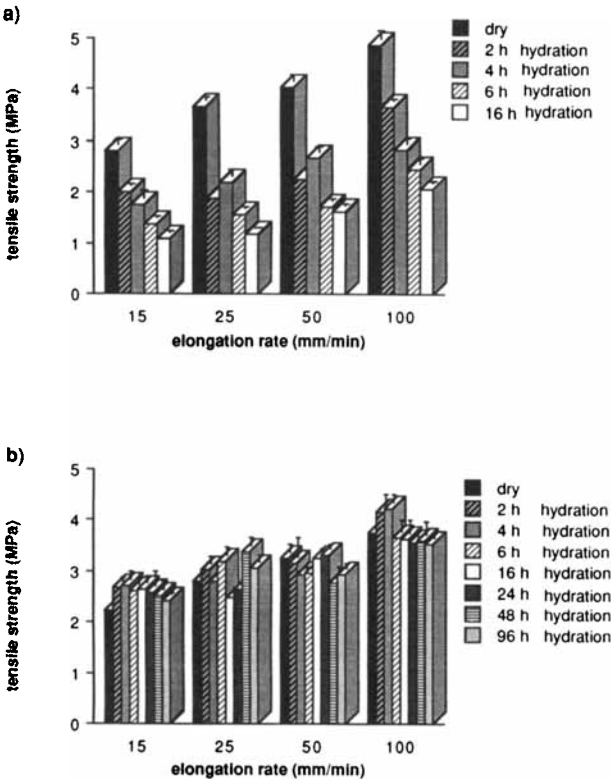

Figure 4. Infuence of elongation rate on the tensile strength of the dressings at different hydration times: (a) regular dressing; (b) nondispersive dressing.

Both regular and nondispersive dressings exhibit, at each hydration level, an increase in tensile strength on increasing elongation rate. This indicates a certain adaptability of both dressings to deformation and can be explained by the elastic nature of the adhesive matrix. Moreover, the increase in tensile strength is less pronounced in the case of nondispersive dressing [Fig. 4(b)], and this can be explained by the more rigid structure of the adhesive matrix in this dressing.

Figures 5(a) and 5(b) illustrate the relationships between the tensile strength values and the degree of hydration at two elongation rates (15 and 100 mm/min). For the regular dressing [Fig. 5(a)] a significant decrease ($p < 0.01$) in tensile strength is observed on increasing water uptake. This result is consistent with that obtained for resistance to penetration [Fig. 3(a)]. The tensile strength values observed for the nondispersive dressing are, on the contrary, independent of the amount of water taken up; this behavior is also in

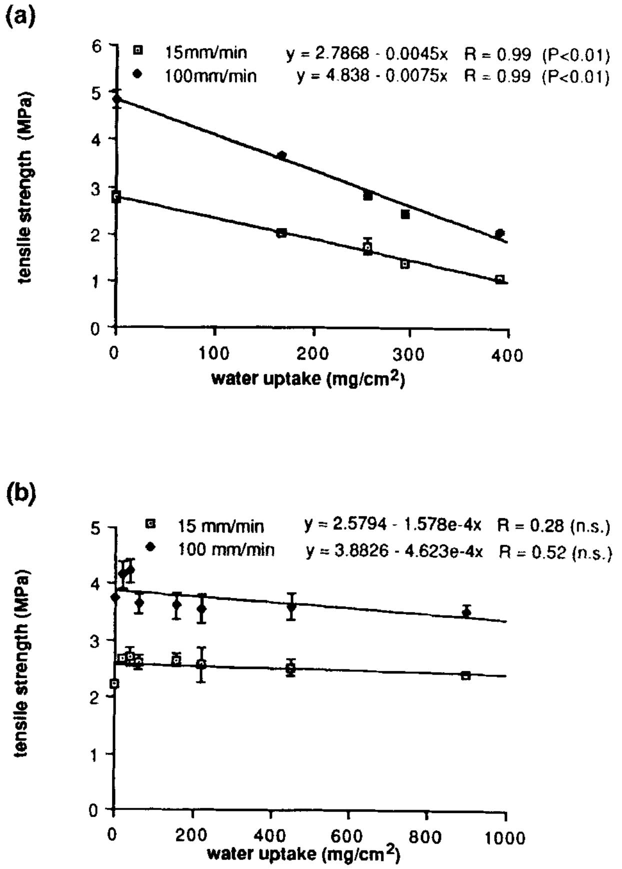

Figure 5. Relationships between tensile strength of the dressings at two elongation rates and degree of hydration: (a) regular dressing; (b) nondispersive dressing.

line with the gel strength values reported above [Fig. 3(b)].

Rheological Data

Figures 6(a) and 6(b) show the influence of the frequency of stress application by the dynamic oscillatory test on the storage or elastic modulus (G') and the loss of viscous modulus (G'') values obtained for the nondispersive dressing at increasing hydration levels. Each value is the mean ($\pm$ *SE*) of three replicates.

A continuous increase of both parameters can be observed: in particular, the increase of the storage modulus G' is more pronounced than that of the loss modulus G''. This can be explained by the increasing elastic, rather than the viscous, characteristics of the material after hydration. This behavior is typical of pressure-sensitive adhesives (10). Similar results were obtained for the regular dressing.

Figures 7(a) and 7(b) show the change in storage modulus G' with increasing hydration for both dressings. For the regular dressing [Fig. 7(a)] a sharp decrease in the storage modulus G' is observed soon after hydration (2 hr). A small amount of water is therefore responsible for a significant reduction of the inherent elastic properties of this dressing; this is accompanied by a reduction in dressing integrity, as suggested by the resistance to compression measurements [Fig. 3(a)].

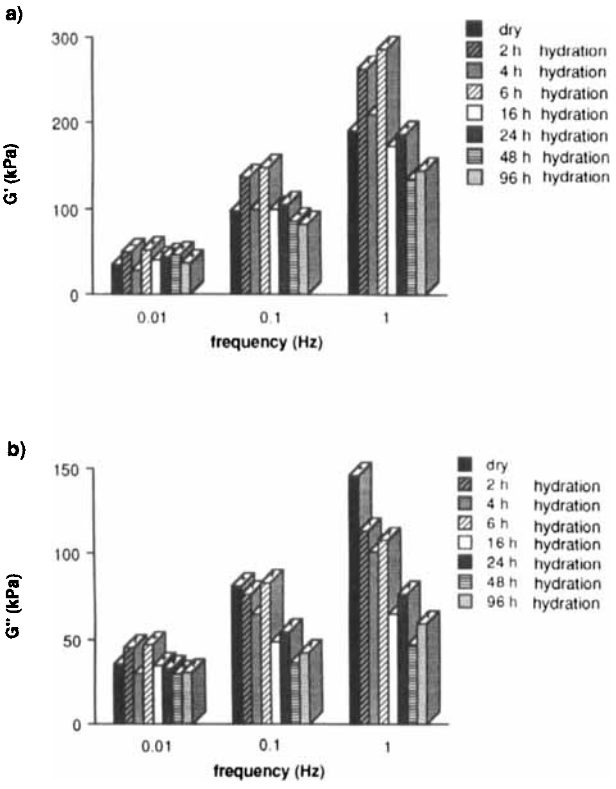

Figure 6. Influence of frequency of stress application on G' and G'' values for the nondispersive dressing.

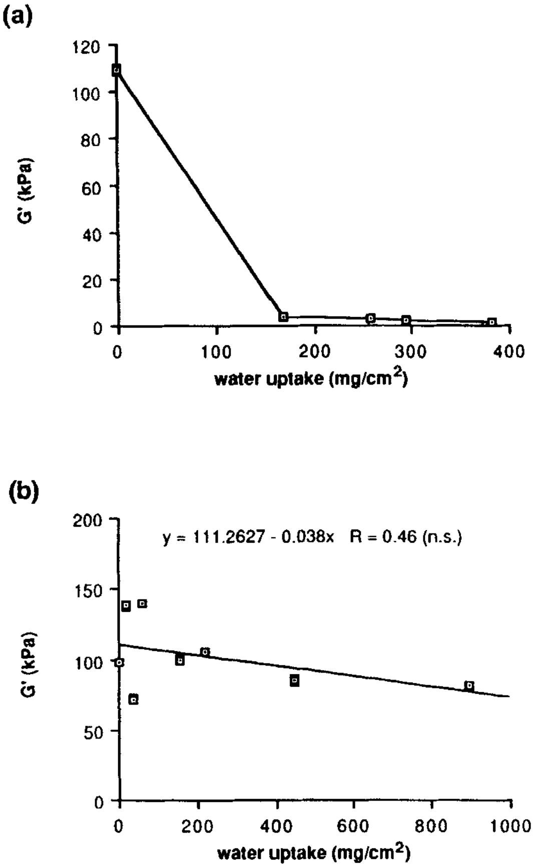

Figure 7. Relationship between the storage modulus value (G') and the hydration level: (a) regular dressing; (b) nondispersive dressing.

In comparison, the storage modulus of the nondispersive dressing [Fig. 7(b)], even after long periods of hydration, remains almost unchanged. This indicates that this dressing maintains its viscoelastic properties, even after hydration, and the dressing maintains its integrity. Similar results were obtained when the dependence of loss modulus (G'') on hydration level was considered for both dressings.

Adhesion Data

Figure 8(a) and 8(b) illustrate the influence of the degree of hydration on the maximum force of detach-

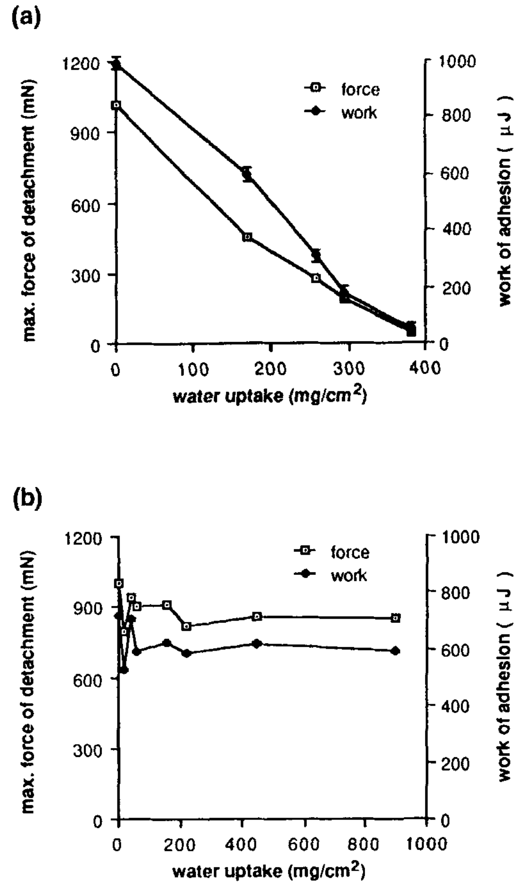

Figure 8. Relationships between maximum force of detachment and work of adhesion and degree of hydration: (a) regular dressing; (b) nondispersive dressing.

ment and on the work of adhesion. The same pattern is observed for both parameters, although they measure different properties. Maximum force of detachment is related to the adhesiveness of the dressing and the work of adhesion is indicative of the viscoelastic properties of bioadhesive gels (11,12).

The significant decrease in maximum force of detachment observed for the regular dressing [Fig. 8(a)] indicates a reduction of the adhesive properties after short time periods (2–4 hr); the parallel decrease in work of adhesion is probably accompanied by an important variation in the viscoelastic properties, concomitant with the loss of integrity of this dressing. For the nondispersive dressing the maximum force of detachment and work of adhesion remain almost constant on increasing degree of hydration [Fig. 8(b)]; this indicates the maintenance of the initial adhesive and viscoelastic properties of the dressing even after long periods of hydration and suggests the use of this dressing in clinical circumstances when it is necessary to increase the duration of application of a single dressing. The dependence of work of adhesion and gel strength values on degree of hydration is summarized in Figs. 9(a) and 9(b) for the two dressings, showing similar trends. The dependence of work of adhesion and of the viscoelastic parameter *G'* on degree of hydration is illustrated in Figs. 10(a) and 10(b). For the regular dressing the decrease of work of adhesion is accompanied by the parallel decrease of gel strength values [Fig. 9(a)] and *G'* values [Fig. 10(a)].

It can be concluded that the increase in degree of hydration produces a softening of the gel layer formed at the interface with the exudating wound, which is accompanied by the occurrence of an irreversible plastic deformation and loss of the elastic properties.

For the nondispersive dressing both mechanical and viscoelastic properties remain practically unchanged after hydration, as is the case for work of adhesion

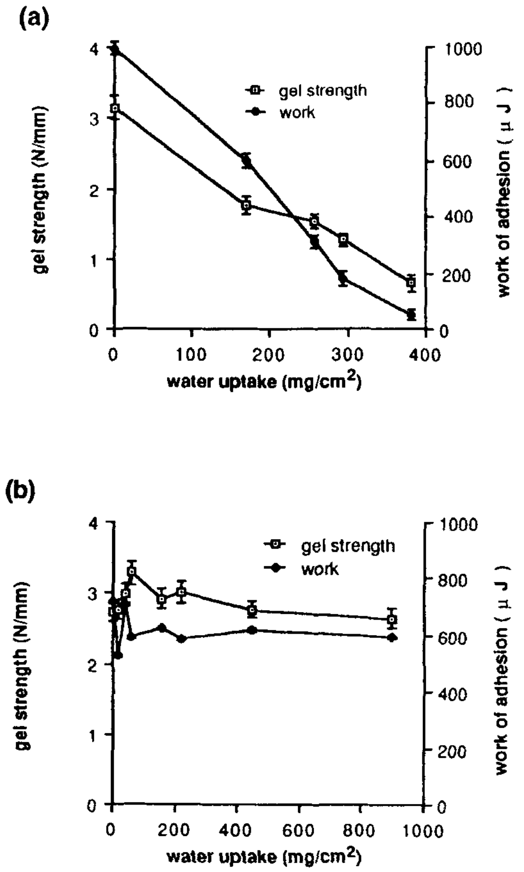

Figure 9. Relationships between gel strength and work of adhesion and degree of hydration: (a) regular dressing; (b) nondispersive dressing.

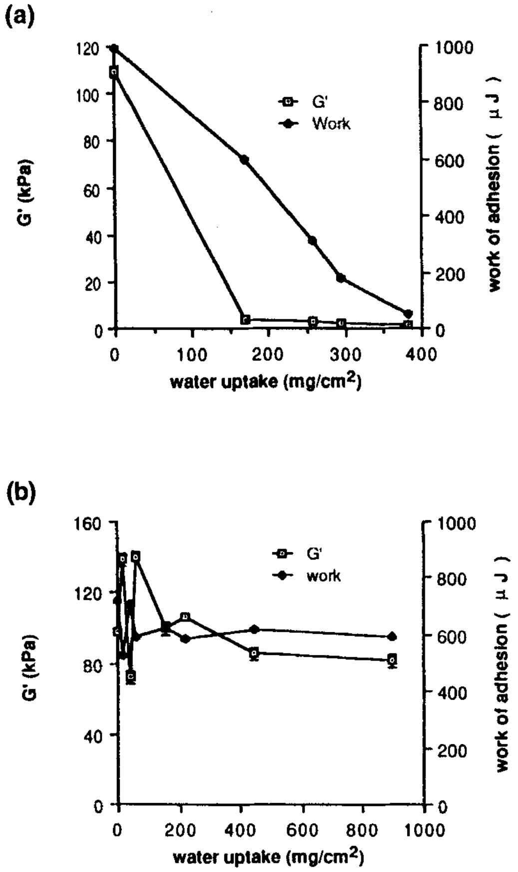

Figure 10. Relationships between *G'* value and work of adhesion and degree of hydration: (a) regular dressing; (b) nondispersive dressing.

[Figs. 9(b) and 10(b)]. This indicates the maintenance of the integrity of this sample, that is, of its viscoelastic and adhesive properties.

CONCLUSIONS

The two dressings showed different hydration characteristics. Regular dressing takes up water rapidly and tends to approach saturation after 12 hr. It undergoes swelling and loss of integrity accompanied by reduction in both rheomechanical and adhesive properties. The nondispersive dressing exhibits a linear water uptake profile over extended periods of hydration up to 4 days. It does not swell and its rheomechanical and adhesive properties remain virtually unchanged after hydration. The difference in performance of the two dressings can be explained by the modified composition of the nondispersive dressing, resulting in a more rigid structure which inhibits complete and rapid liquid uptake.

The comparative evaluation of the results obtained with the various methods points to the relevance of mechanical and viscoelastic properties to functionality of dressings.

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