# Retention of propanal and diacetyl in experimental resins

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The degree of evaporation from experimental resins containing 0–40 mol% propanal or diacetyl was determined over a 6-month period at 60 °C. From the results the maximum evaporation possible, $M_\infty$, was calculated for each resin and was found to vary between 0.28% and 7.51% by weight. At low contents of propanal or diacetyl, $M_\infty$ remained unchanged as compared with the control resins without additive. At higher contents of additive, $M_\infty$ increased significantly. In resins based on BisGMA and TEGDMA, propanal was retained to a lesser extent than diacetyl. In resins based on UEDMA and HEMA, propanal was retained to a greater extent than diacetyl. This study confirms that propanal and diacetyl become bound in the polymer structure, and theories as to the reaction mechanisms are presented. ☐ *Aldehyde; dental materials; evaporation; ketone*

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Addition of the aldehyde propanal (propionaldehyde) or the diketone diacetyl (2,3-butanedione) to methacrylate resins has been shown to increase significantly the conversion of the resins and to improve the mechanical properties and in vitro wear resistance of resin composites (1–7). The extent of conversion continuously increased as the content of propanal or diacetyl increased from 0 to 40 mol% (4, 5). Strength, elastic modulus, and resistance to in vitro wear improved up to a content of 24–32 mol% propanal or diacetyl, above which the properties remained unchanged (1, 3, 7). Hardness, on the other hand, decreased at high contents of propanal or diacetyl (7). The decrease in hardness and the lack of an additional effect on strength, elastic modulus, and in vitro wear found at high contents of propanal or diacetyl were thought to be the result of an excess of propanal or diacetyl, which had a softening effect on the resin. An excess of propanal or diacetyl implies a risk of leaching and evaporation of these rather volatile and relatively toxic components, thus reducing the biocompatibility and consequently the usefulness of resins containing propanal or diacetyl.

The present study was undertaken in an attempt to evaluate the clinical utility of resins containing either propanal or diacetyl. This was done by determining to what extent propanal and diacetyl are retained in photopolymerized methacrylate resins.

## Materials and methods

The resins used in this investigation were prepared from the compounds listed in Table 1; their compositions (in mol%) are given in Table 2. To make the materials light-curing, 0.2% w/w of CQ and 0.2% w/w of CEMA were dissolved in each of the monomer mixtures. The resins were identical to those studied in earlier work (1, 2, 4, 5, 7).

A brass mold (diameter, 15 mm; height, 1 mm) was filled with monomer (195 µl), covered on both sides with a clear matrix strip, and irradiated with a Visilux 2 unit (3M Company, St. Paul, Minn., USA) for 80 sec on each side. The specimen was separated from the mold and immediately weighed on an analytic balance to the nearest 0.01 mg (Mettler H54, Mettler Instrumente AG, Greifensee-Zürich, Switzerland). This weight served as base-line value. Three specimens were produced from each of the 22 monomer mixtures. With the aid of plastic holders, the 66 specimens were placed vertically in an oven at 60 °C and weighed after 1 week, 1 month, 3 months, and 6 months. After each weighing, the weight was converted to a percentage of the base-line value, and the mean value and standard deviation of the three values of percentage weight loss of each resin was computed. The statistical treatment of the results involved one-way analysis of variance (8) and the Newman–Keuls multiple range test (9).

## Results

The mean values and standard deviations of the quantity of substance evaporated from the 22 resins are shown in Table 3 for each of the 4 measuring times. A1–A12 indicates the quantities evaporated from propanal-containing specimens, and K1–K12 the quantities evaporated from diacetyl-containing specimens. To improve visualization of the effect of propanal

Table 1. Materials used for preparation of resins

| Compound | Acronym | Supplier |
|---|---|---|
| Bisphenol-A-glycidyl dimethacrylate | BisGMA | Röhm Chem. Fabrik, Darmstadt, Germany |
| Triethylene glycol dimethacrylate | TEGDMA | Aldrich-Chemie, Steinheim, Germany |
| Urethane dimethacrylate* | UEDMA | Ivoclar AG, Schaan, Liechtenstein |
| 2-Hydroxyethyl methacrylate | HEMA | Merck-Schuchardt, Munich, Germany |
| Camphorquinone | CQ | EGA-Chemie, Albuch, Germany |
| *N,N*-cyanoethylmethylaniline | CEMA | Ivoclar AG |
| Propanal | PAL | Merck-Schuchardt |
| Diacetyl | DIAC | Merck-Schuchardt |

* From Ruyter (22).

and diacetyl, the values of the control resins (A1 = K1 and A7 = K7) are listed for the propanal-containing resins and for the diacetyl-containing resins.

For each of the 22 resins an estimation was made of the quantity of substance evaporated at infinite time—that is, the maximum evaporation loss possible. This estimation was based on knowledge of the relationship between total quantity of substance $M_t$ evaporated and time t. Thus, in the initial stages of evaporation $M_t$ is proportional to $t^{1/2}$; (10). Furthermore, as time moves towards infinity, $M_t$ must approach a final value $M_\infty$. A formula that accounts for both tendencies is $1/M_t = a + b\,(1/t^{1/2})$. Linear regression analysis using $1/M_t$ and $1/t^{1/2}$ as variables showed in all cases significant coefficients of correlation. The coefficients of correlation varied between 0.732 and 0.999, with a mean value of 0.951 and a standard deviation of 0.063. An example of a regression plot is shown in Fig. 1 (K10). Determination of 'a' in the above-mentioned formula enables the calculation of the maximum evaporation $M_\infty$, which equals $1/a$. $M_\infty$ is listed in Table 3 and depicted in Fig. 2.

The 22 mean values of maximum evaporation $M_\infty$ differed with statistical significance ($P < 0.005$). Differences between the mean values were then analyzed by the Newman–Keuls multiple range test, and in Table 3 identical lettering designates mean values of $M_\infty$ with no statistically significant difference ($P > 0.05$). There was no significant difference in $M_\infty$ between the control resin based on BisGMA and TEGDMA and the control resin based on UEDMA and HEMA. For resins containing either propanal or diacetyl, $M_\infty$ increased with increased content of additive. At a propanal content of 24 mol% or more, $M_\infty$ was higher for resins based on BisGMA and TEGDMA than for the corresponding resins based on UEDMA and HEMA. With regard to diacetyl-containing resins, $M_\infty$ was higher for UEDMA/HEMA-based resins than for

Table 2. Composition (mol%) of resins A1–A12 and K1–K12

| Code | BisGMA | TEGDMA | UEDMA | HEMA | PAL | DIAC |
|---|---|---|---|---|---|---|
| A1 = K1 | 25 | 75 | | | | |
| A2 | 23 | 69 | | | 8 | |
| A3 | 21 | 63 | | | 16 | |
| A4 | 19 | 57 | | | 24 | |
| A5 | 17 | 51 | | | 32 | |
| A6 | 15 | 45 | | | 40 | |
| A7 = K7 | | | 75 | 25 | | |
| A8 | | | 69 | 23 | 8 | |
| A9 | | | 63 | 21 | 16 | |
| A10 | | | 57 | 19 | 24 | |
| A11 | | | 51 | 17 | 32 | |
| A12 | | | 45 | 15 | 40 | |
| K1 = A1 | 25 | 75 | | | | |
| K2 | 23 | 69 | | | | 8 |
| K3 | 21 | 63 | | | | 16 |
| K4 | 19 | 57 | | | | 24 |
| K5 | 17 | 51 | | | | 32 |
| K6 | 15 | 45 | | | | 40 |
| K7 = A7 | | | 75 | 25 | | |
| K8 | | | 69 | 23 | | 8 |
| K9 | | | 63 | 21 | | 16 |
| K10 | | | 57 | 19 | | 24 |
| K11 | | | 51 | 17 | | 32 |
| K12 | | | 45 | 15 | | 40 |

Table 3. Mean values and standard deviations of evaporated amount $M_t$ in percentage at the four measuring times and of the estimated maximum evaporation $M_\infty$. $M_\infty$ values with same lettering were not significantly different

| Experimental composite | $M_t$ (%) ($t$ = 1 week) | $M_t$ (%) ($t$ = 1 month) | $M_t$ (%) ($t$ = 3 months) | $M_t$ (%) ($t$ = 6 months) | $M_t$ (%) ($t = \infty$) |
|---|---|---|---|---|---|
| A1 = K1 | 0.36 ± 0.03 | 0.42 ± 0.01 | 0.48 ± 0.05 | 0.52 ± 0.04 | $^{ab}$0.56 ± 0.02 |
| A2 | 0.32 ± 0.05 | 0.36 ± 0.02 | 0.47 ± 0.04 | 0.49 ± 0.04 | $^{ab}$0.55 ± 0.06 |
| A3 | 0.40 ± 0.01 | 0.41 ± 0.02 | 0.51 ± 0.01 | 0.56 ± 0.03 | $^{ab}$0.57 ± 0.07 |
| A4 | 0.52 ± 0.02 | 0.64 ± 0.01 | 0.89 ± 0.07 | 1.04 ± 0.01 | $^{c}$1.24 ± 0.21 |
| A5 | 0.80 ± 0.03 | 1.12 ± 0.03 | 1.62 ± 0.05 | 1.85 ± 0.03 | $^{e}$2.62 ± 0.35 |
| A6 | 1.54 ± 0.16 | 2.26 ± 0.23 | 2.91 ± 0.26 | 3.35 ± 0.28 | $^{g}$4.63 ± 0.16 |
| A7 = K7 | 0.28 ± 0.04 | 0.32 ± 0.07 | 0.37 ± 0.03 | 0.42 ± 0.06 | $^{ab}$0.44 ± 0.03 |
| A8 | 0.13 ± 0.03 | 0.15 ± 0.03 | 0.21 ± 0.03 | 0.25 ± 0.03 | $^{a}$0.28 ± 0.06 |
| A9 | 0.29 ± 0.02 | 0.34 ± 0.02 | 0.41 ± 0.01 | 0.51 ± 0.02 | $^{ab}$0.54 ± 0.07 |
| A10 | 0.32 ± 0.04 | 0.41 ± 0.03 | 0.54 ± 0.04 | 0.69 ± 0.05 | $^{ab}$0.81 ± 0.14 |
| A11 | 0.43 ± 0.01 | 0.58 ± 0.06 | 0.84 ± 0.05 | 1.04 ± 0.05 | $^{c}$1.39 ± 0.27 |
| A12 | 0.80 ± 0.01 | 1.29 ± 0.04 | 1.83 ± 0.11 | 1.99 ± 0.08 | $^{f}$3.44 ± 0.21 |
| K1 = A1 | 0.36 ± 0.03 | 0.42 ± 0.01 | 0.48 ± 0.05 | 0.52 ± 0.04 | $^{ab}$0.56 ± 0.02 |
| K2 | 0.23 ± 0.02 | 0.27 ± 0.04 | 0.29 ± 0.02 | 0.34 ± 0.03 | $^{a}$0.35 ± 0.02 |
| K3 | 0.36 ± 0.03 | 0.43 ± 0.04 | 0.53 ± 0.03 | 0.56 ± 0.04 | $^{ab}$0.64 ± 0.04 |
| K4 | 0.69 ± 0.02 | 0.78 ± 0.02 | 0.85 ± 0.01 | 0.91 ± 0.01 | $^{bc}$0.96 ± 0.03 |
| K5 | 1.43 ± 0.27 | 1.63 ± 0.28 | 1.69 ± 0.29 | 1.77 ± 0.29 | $^{d}$1.86 ± 0.02 |
| K6 | 3.63 ± 0.46 | 3.93 ± 0.50 | 4.03 ± 0.51 | 4.11 ± 0.51 | $^{g}$4.25 ± 0.01 |
| K7 = A7 | 0.28 ± 0.04 | 0.32 ± 0.07 | 0.37 ± 0.03 | 0.42 ± 0.06 | $^{ab}$0.44 ± 0.03 |
| K8 | 0.08 ± 0.05 | 0.15 ± 0.06 | 0.20 ± 0.05 | 0.24 ± 0.06 | $^{ab}$0.54 ± 0.10 |
| K9 | 0.08 ± 0.00 | 0.17 ± 0.01 | 0.22 ± 0.01 | 0.32 ± 0.01 | $^{c}$1.31 ± 0.93 |
| K10 | 0.21 ± 0.01 | 0.37 ± 0.00 | 0.51 ± 0.02 | 0.65 ± 0.02 | $^{c}$1.34 ± 0.10 |
| K11 | 0.75 ± 0.07 | 1.30 ± 0.12 | 1.84 ± 0.15 | 2.15 ± 0.17 | $^{g}$4.34 ± 0.20 |
| K12 | 2.60 ± 0.31 | 3.97 ± 0.35 | 4.89 ± 0.33 | 5.31 ± 0.34 | $^{h}$7.51 ± 0.29 |

BisGMA/TEGDMA-based resins at a diacetyl content of 16 mol%, 32 mol%, or 40 mol%.

## Discussion

For the 22 experimental resins investigated, the estimated maximum quantity of substance $M_\infty$ which will evaporate varied between 0.28 wt% and 7.51 wt%. The BisGMA/TEGDMA and the UEDMA/HEMA control resins (A1 = K1 and A7 = K7) showed statistically similar $M_\infty$ values of 0.56% and 0.44%, respectively. These values of volatility are in agreement with values of elution of leachable components. Thus, elution of leachable components from six proprietary resin

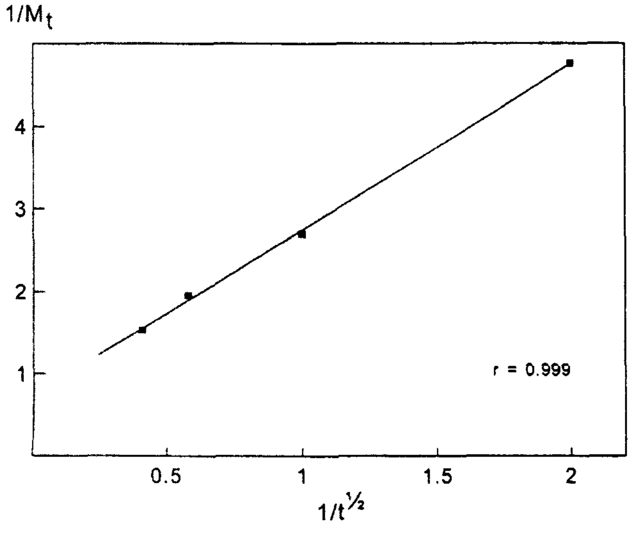

Fig. 1. Linear relationship between $1/t^{1/2}$ and $1/M_t$ for K10 resin composite ($r$ = 0.999, $P < 0.001$). M = amount of substance evaporated at a given time, t = time. The regression line intercepts the ordinate at 0.745 (= a). The maximum evaporation $M_\infty$ is calculated as 1/a = 1.34.

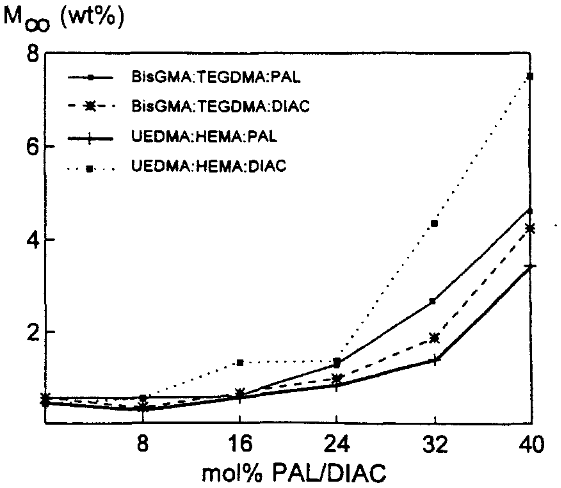

Fig. 2. Relationship between content (mol%) of propanal (PAL) or diacetyl (DIAC) and mean value of maximum evaporation, $M_\infty$ (wt%). The standard deviations are listed in Table 3, as are the mean values. See Table 1 for explanation of the acronyms.

composites into water was reported to vary between 0.25% and 3.0% of the initial weight of the polymerizable resin (11). Elution was determined after a 48-h period, within which 95% of the elutable species will be extracted (12). Other studies of the elution of composite components have shown that virtually all components in dental resin composites may be leached into solution, with the vast majority of the eluted components being monomers (13–15). Consequently, the main part of the evaporated quantity of substance from the present resin control materials may be expected to consist of residual TEGDMA monomer or oligomer (A1 = K1) or of HEMA monomer or oligomer (A7 = K7), since the high molecular weight monomers BisGMA and UEDMA are much less volatile.

The maximum quantity of evaporation possible ($M_\infty$) did not change as a result of addition of low levels of propanal or diacetyl; depending on the experimental series, between 8 mol% and 24 mol% propanal or diacetyl could be added without a significant change in $M_\infty$. Higher contents of additive in each of the four series meant a significant increase in $M_\infty$. The fact that propanal or diacetyl could be added to the resins without an increase in $M_\infty$ is indicative of the additive having been bound in the polymer to some extent. In earlier studies aldehydes and diketones were hypothesized to have a cross-linking ability and thus to be able to react with and connect pendant or backbone functional groups present in dental polymers (1–3). The cross-linking reactions proposed were nucleophilic mechanisms, which normally require a strong base as catalyst to take place with appreciable rate. The experimental monomers are not basic, but the photoreductant used is a base, although a weak one. However, subsequent studies of the degree of conversion of methacrylate double bonds found significant increases in conversion as the result of propanal or diacetyl addition, and it was concluded that the significant improvements in mechanical properties brought about by propanal or diacetyl addition were caused mainly by increases in the degree of conversion rather than by an enhanced degree of cross-linking between functional groups other than methacrylate double bonds (4, 5). The positive effect on mechanical properties of propanal or diacetyl addition has also been suggested to be the result of free radical mechanisms (2, 3, 5). Ketones undergo keto-enol tautomerism, and certain diketones may exist in the enol form to an appreciable extent. This form, containing a double bond, was presumed to be able to participate in the polymerization reactions of the methacrylate monomers and lead to an increased degree of conversion (4). However, as the effect of

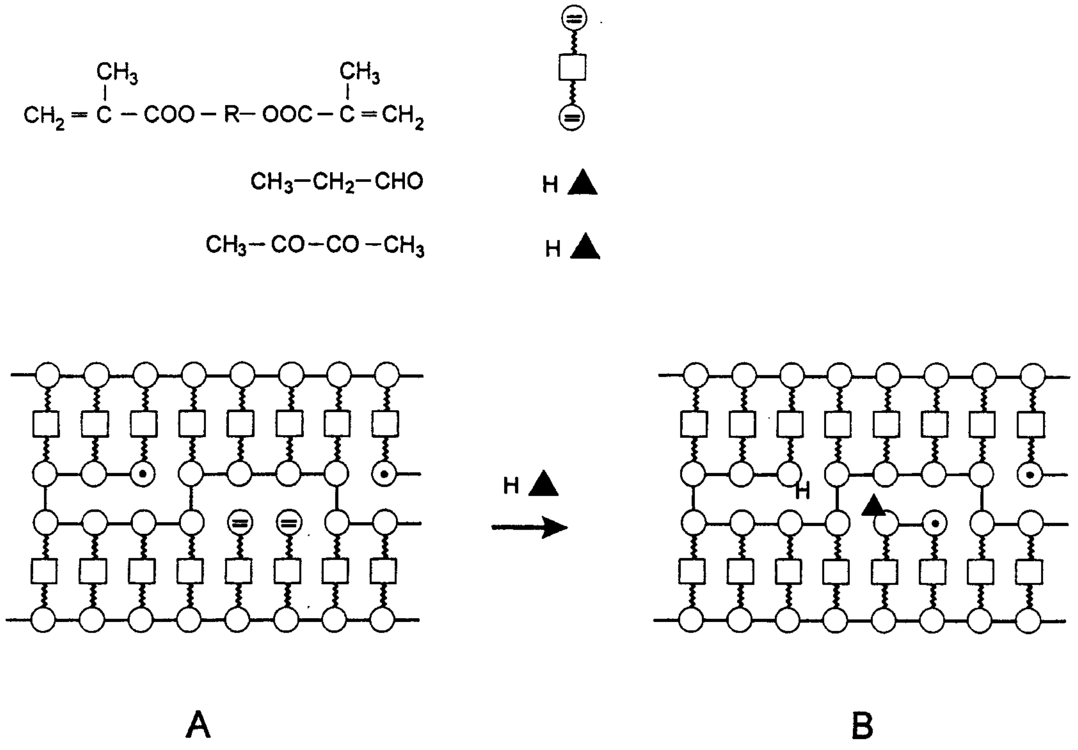

Fig. 3. Hypothesized chain transfer reaction between propanal or diacetyl and an unreacted double bond present in a growing polymer and situated adjacent to another unreacted double bond. 3A) Simplified representation of the three-dimensional structure of a growing polymer of dimethacrylate monomers with cross-linking (—), free radicals (⊙), and unreacted double bonds (⊜). 3B) A hydrogen from the chain transfer agent has reacted with a free radical of the growing polymer. By means of diffusion, the chain transfer radical has then reached and reacted with one of the two unreacted double bonds, and a crosslink has been formed to the adjacent unreacted double bond.

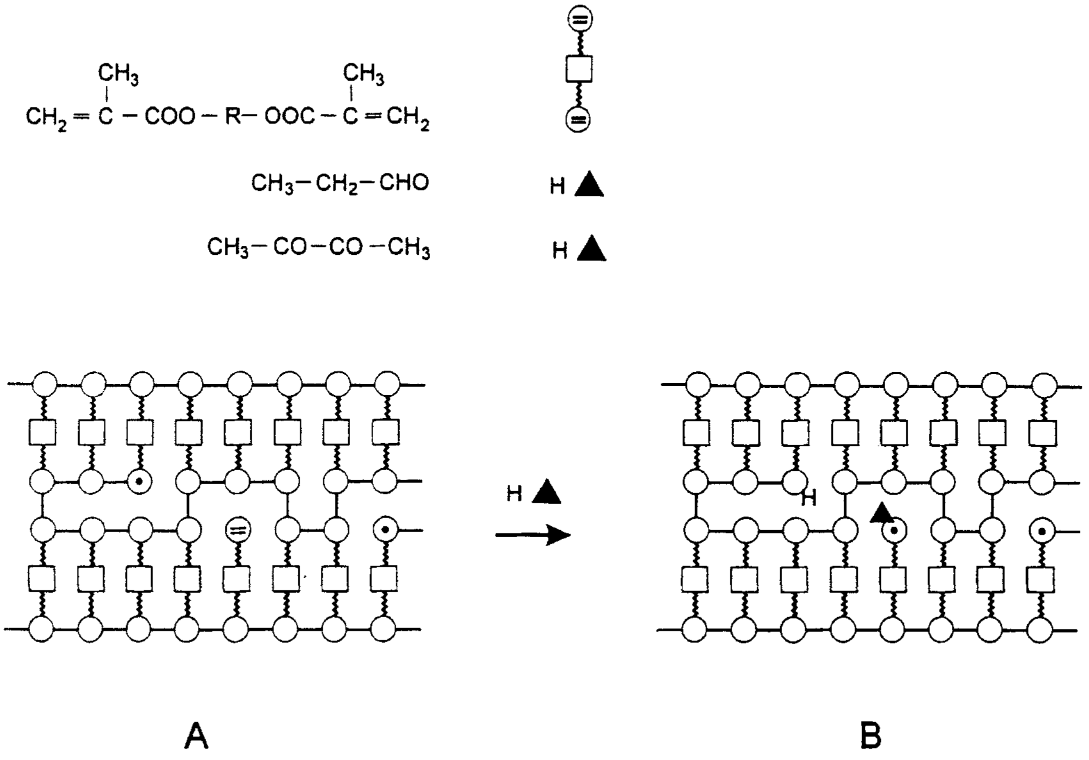

Fig. 4. Hypothesized chain transfer reaction between propanal or diacetyl and a singly unreacted double bond present in a growing polymer. 4A) Simplified representation of the three-dimensional structure of a growing polymer of dimethacrylate monomers with crosslinking (—), free radicals (⊙), and unreacted double bonds (⊜). 4B) A hydrogen from the chain transfer agent has reacted with a free radical of the growing polymer. By means of diffusion, the chain transfer radical has then reached and reacted with the unreacted double bond.

propanal on degree of conversion has been found to be as pronounced as that of diacetyl (5), keto-enol tautomerism does not seem a likely explanation. Further, when irradiated, ketones were noted to be able to form free radicals, which may either initiate polymerization or, as may aldehydes, add to the carbon–carbon double bonds of the methacrylate groups in the presence of a photoreducing agent, thus increasing the degree of conversion (4, 5). The fact that the degree of conversion of chemically cured propanal-containing resins was as high as that of light-cured resins indicated that propanal does not react only in the presence of free radical photoinitiators (6). Although the conversion of diacetyl-containing, light-cured resins was significantly higher than that of the chemically cured corresponding resins, which suggested a certain photoinitiating function, the primary mode of effect of diacetyl was argued to be identical to that of propanal (6).

Aldehydes can act as chain transfer agents (16, 17), and both propanal and diacetyl in the present experimental resins have been suggested to act in this exact manner (6). Fig. 3 shows a theory of how propanal and diacetyl exert their effect as chain transfer agents and promote an increase in the degree of conversion and the extent of cross-linking. In a growing polymer a free radical on the polymer reacts with the chain transfer agent by hydrogen abstraction. By way of diffusion, the new radical consisting of the remaining chain transfer molecule can reach and then react with a double bond, which, because of steric hindrance, would otherwise not have reacted. Furthermore, in the early stages of polymerization, characterized by substantial unsaturation, a cross-link will be formed to an adjacent unreacted double bond. Even though the presence of the reacted chain transfer molecule in the network has a destabilizing effect which tends to weaken the polymer, the net result of the reaction on hardness and strength is positive because of increased cross-linking. The evaporated substance from experimental resins in which these chain transfer reactions have taken place would then be expected to consist of residual TEGDMA or HEMA monomer and of unreacted propanal or diacetyl.

As mentioned above, enhanced and significantly differing values of $M_{\infty}$ were measured in resins with high contents of additive. This presumably indicates the presence of an excess of propanal or diacetyl and implies that the evaporated quantity of substance from these resins to a higher extent consists of propanal or diacetyl and to a much smaller extent of residual TEGDMA monomer or oligomer (A1 = K1) or of HEMA monomer or oligomer (A7 = K7).

Assuming that all evaporated quantity of substance was either propanal or diacetyl and not monomer or other resin components, calculations were made of the fraction of substance which can evaporate from specimens of resins containing from 8 mol% to 40 mol% propanal or diacetyl. The calculations showed that the fraction of the added propanal which can evaporate from propanal-containing BisGMA/TEGDMA resins was higher (0.18–0.46) than the fraction of the added diacetyl which can evaporate from corresponding diacetyl-containing BisGMA/TEGDMA resins (0.13–0.30). As for the UEDMA/HEMA-based resins, the fraction of the added propanal which can evaporate from propanal-containing resins was less (0.18–0.38) than the fraction of diacetyl which can evaporate from diacetyl-containing resins (0.20–0.58). At present no definite explanation can be offered as to the difference in retention of propanal and diacetyl dependent on monomer composition, although the differences are thought in some way to reflect differences in chain transfer constants of propanal and diacetyl (6).

As mentioned in the introduction, a low content of propanal or diacetyl improved the hardness of the experimental resins, whereas a high content of propanal or diacetyl impaired the hardness (7). As $M_\infty$ showed a similar pattern, a two-dimensional correlation analysis was performed between $M_\infty$ and Wallace indentation hardness for each of the four series of experimental resins. The coefficients of correlation varied between 0.69 and 0.89 and differed from zero at $P < 0.0005$. The fact that a high content of additive led to increased evaporation, decreased hardness, and increased degree of conversion but to unchanged strength is an apparent disparity, an explanation of which is offered in Fig. 4. The figure shows a resin in a late stage of polymerization but still with unreacted chain transfer agent. In this case, some remaining unreacted double bonds may be expected to be situated singly rather than in pairs or groups as they do in resins with less conversion. The destabilization caused by the presence of the chain transfer agent will not be accompanied by additional cross-linking but only by an increased degree of conversion. However, this increase is smaller than the one obtained when unreacted double bonds appear in pairs or groups as in resins in earlier stages of polymerization—that is, in resins with less conversion (Fig. 3). The net result in resins with a high content of propanal or diacetyl is that the degree of conversion is increased, the strength is maintained owing to unchanged degree of cross-linking, while hardness is decreased due to destabilization and softening of the polymer by the chain transfer molecules, bound or unbound.

As mentioned previously, proprietary resin composites leach several components, mainly residual monomer, into aqueous media (13–15). The potential impact that this may have on the biocompatibility of the material has been of great concern, and several studies have investigated the elution of unbound molecules from resin composites. Most of the unbound components have been found to be released with 24 h (12). Dental composites have been shown to have significant cytotoxicity in cell culture (18, 19). Pulp studies, on the other hand, have shown lack of significant pulpal irritation after the placement of properly sealed resin composite fillings (20). This poor correlation between in vitro cytotoxicity evaluations and the in vivo pulp toxicity tests has been explained by the rapid release of unbound components, and it was concluded that resin composites do not seem to provide a chronic source of unreacted monomer to the pulp or other oral tissue (12).

For a particular resin, the quantity of monomer leached will depend on the degree of conversion (21). Addition of small quantities of propanal or diacetyl will increase the degree of resin conversion, and the quantity of unreacted monomer which may evaporate or leach will be decreased. However, a quantity of residual propanal or diacetyl will also evaporate or leach. The $LD_{50}$ value of dimethacrylates may be expected to be a little higher than that of methyl methacrylate, which in rats has been determined as 7.3 g/kg (National Register of Chemical Substances and Products, National Institute of Occupational Health, Denmark; Personal communication). Although somewhat lower, the corresponding values for propanal and diacetyl (1.4 g/kg and 1.6 g/kg) are within the same order of magnitude (National Register of Chemical Substances and Products, National Institute of Occupational Health, Denmark; Personal communication). It is concluded that the biocompatibility of resin composites with a content of up to 24 mol% propanal or diacetyl may be similar to that of the present proprietary resin composites.

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