

The Level of Residual Monomer in Acrylic Denture Base Materials

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Abstract: The aim of this experimental study, is to perform quantitative analysis of the residual monomer in Acropars and Meliodent heat-cured acrylic resins by means of gas chromatography method and to compare these results with ISO standard. In this research, 12 disks (50×3 mm) were prepared by Acropars and Meliodent acrylic resins. The residual monomer was extracted by methanol and gas chromatography utilized to determine the residual monomer content of samples. There was no residual monomer pick in none of the chromatograms obtained from 0.4 and 2 g of Acropars and Meliodent samples. Acropars acrylic resin complied with the requirements of ISO 1567 regarding residual monomer.

Key words: Residual monomer, methyl methacrylate, heat-cured acrylic resin, gas chromatography

INTRODUCTION

Acrylic resins have been used as denture bases for more than 60 years (Mc Cabe and Basker, 1976). It is important to determine the residual monomer content of the acrylic resins, as this property influences the allergy susceptibility of acrylic dentures (Craig *et al.*, 2000). The released residual monomer is the primary cause of this irritation (Craig, 2002; Faltermeier *et al.*, 2007). On the other hand, it has been proven that there is a relationship between residual monomer and water sorption. If residual monomer is present, less monomer conversion occurs and may result in increasing sorption and solubility (Jagger, 1978; Umemoto and Kurata, 1997). The absorption of water leads to plasticization of the resin, making it more flexible and resilient (Fletcher *et al.*, 1983; Pfeiffer and Rosenbauer, 2004). According to ISO 1567, the maximum residual monomer content of denture base materials should not exceed 2.2% wt (Pfeiffer and Rosenbauer, 2004).

Attempts have been made to reduce the residual monomer content of acrylic resins by using thermoplastic and microwave polymerization rather than heat polymerization (Blagojevic and Murphy, 1999; Miettinen *et al.*, 1999; Kedjarune *et al.*, 1999; Alves *et al.*, 2007). An increase in polymerization temperature and time was accompanied by a decrease in residual monomer content (Jagger, 1978; Miettinen and Vallittu, 1997; Dogan *et al.*, 1995; Wong *et al.*, 1999;

Vallittu *et al.*, 1998; Harrison and Huggett, 1992; Kalipcilar *et al.*, 1991). Tsuchiya *et al.* (1994), Vallittu *et al.* (1995) and Shim and Watts (1999) have demonstrated that the residual monomer content could be lowered if the denture was stored in water after processing.

Another significant factor for residual monomer release is the surface condition of the denture base resin. Specimens polished to high luster demonstrated significantly reduced monomer release in comparison with unpolished one (Pfeiffer and Rosenbauer, 2004; Vallittu, 1996). Sample thickness, drill speed (Fletcher *et al.*, 1983), measuring method (Rose *et al.*, 2000; Urban *et al.*, 2007; Duray and Gilbert, 1997; Azzari *et al.*, 2003; Austin and Basker, 1980; Imazato *et al.*, 2001) and usage period of dentures (Sadamori and Kotani, 1992) are the other important factors which may alter the residual monomer content of acrylic dentures.

Acropars (Marlic Co. Tehran. Iran) is an Iranian heat polymerized acrylic resin which has become available in recent years. In this study, Acropars is investigated for the residual monomer and the results are compared with the Meliodent (Bayer dental, Bayer UK limited) and ISO standard.

MATERIALS AND METHODS

In this laboratory research, twelve copper disks (50×3 mm) were invested in conventional denture flasks

in dental stone (Hinritz stone, Ernst Hinrichs GmbH, Germany). Six samples were prepared by each of the acrylic resin. All samples were processed according to the manufacturer's recommendations. The residual monomer was analyzed immediately after processing.

A standard calibration curve (Fig. 1) was obtained by plotting peak area of 3 standard solutions containing known concentrations (0.01, 0.02 and 0.03%) of the monomer in methanol. Each acrylic specimen was put into the separated flasks. Then 30 mL of distilled water was added. The flasks were stored in a dark place for 72 h. A 3 mm twisted drill was used. About 0.4 g of drill cuttings was collected and poured into covered glass test tubes. Each tube contained 10 mL of methanol (Merck Co. Germany). The test tubes were put on the vortex apparatus (Dena Co., Germany) for one minute. All the tubes were kept in Bain Mary (GFL Co., England) at 65°C for 7 h. Gas chromatography analysis was carried out on a Perkin-Elmer 8500 gas chromatograph with FID detector and a BP-1 capillary column (39×0.25 mm, film thickness 0.25 µm). The carrier gas was nitrogen with a flow rate of 2 mL min⁻¹, the oven temperature was 60°C and then increased at a rate of 24°C min⁻¹ until reached to the temperature of 280°C, injector temperatures were set at 275°C and detector temperatures were set at 280°C.

Confirmation of peak identity was effected by co-chromatography with standards. The quantification of monomer was computed from the GC peak areas obtained using standard curve.

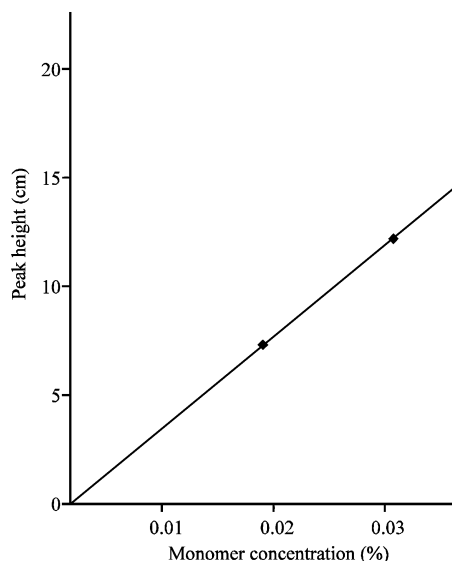


Fig. 1: GC calibration curve for mono methyl methacrylate in methanol

To ensure the reliability of the results, 2 g of sample drill cuttings was used in the second test. All the procedures were repeated the same as the first procedures.

The extracted monomer of samples was calculated by measuring their peak area.

RESULTS AND DISCUSSION

Chromatograms corresponding to the injection of 0.1 (A), 0.2 (B) and 0.3 µL (C) of MMA to 1 cc methanol are shown in Fig. 2.

As shown in Fig. 2 there is no detectable peak of monomer in 0.01 concentrations, but in 0.02 and 0.03 concentrations, the monomer peaks are recorded in 4.5 and 4.16 min, respectively.

Figure 3 and 4 display the chromatograms from 0.4 and 2 g of Acropars (A) and Meliodent (B) acrylic resins. The monomer peak was not detected in none of these samples.

Acrylic resins possess many desirable properties, but it has been known that the acrylic denture bases contain residual monomer which may cause some side effects such as hypersensitivity of oral tissues and altered color stability and mechanical characteristics of denture bases (Mc Cabe and Basker, 1976; Craig *et al.*, 2000; Craig, 2002; Faltermeier *et al.*, 2007; Jagger, 1978; Umemoto and Kurata, 1997; Fletcher *et al.*, 1983; Pfeiffer and Rosenbauer, 2004; Dogan *et al.*, 1995).

Many studies have found that substances leached out from acrylic resin can cause irritation of oral tissue, inflammation, or even an allergic reaction (Mc Cabe and Basker, 1976; Craig *et al.*, 2000; Craig, 2002; Faltermeier *et al.*, 2007).

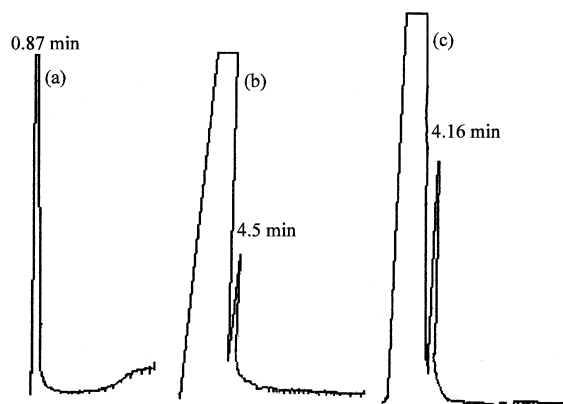


Fig. 2: Chromatograms for (a): 0.01, (b): 0.02 and (c): 0.03 concentration of MMA in methanol

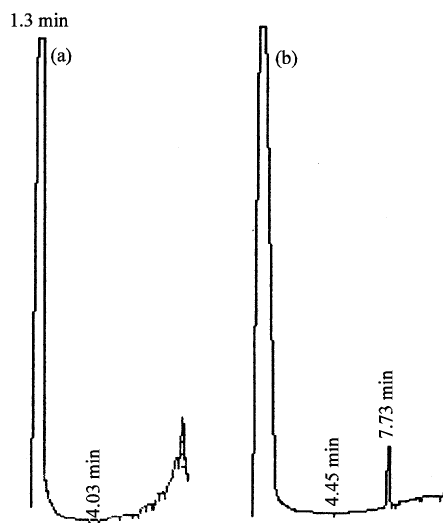


Fig. 3: Chromatograms for 0.4 g of (a): Acropars and (b): Meliodent samples

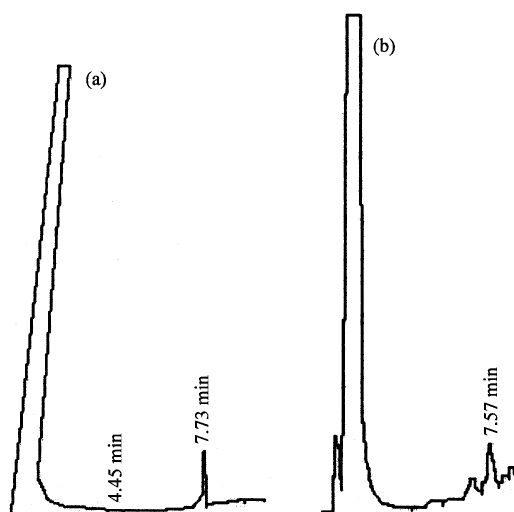


Fig. 4: Chromatograms for 2 g of (a): Acropars and (b): Meliodent samples

On the other hand, an acrylic resin which contains high levels of residual monomer may exhibit low color stability and mechanical strength (Faltermeier *et al.*, 2007; Dogan *et al.*, 1995). The decrease in mechanical properties is due to the loss of residual monomer, which leaches out into either water or saliva and to the absorption of water, which leads to plasticization of the resin, making it more flexible and resilient (Jagger, 1978; Umemoto and Kurata, 1997; Fletcher *et al.*, 1983; Pfeiffer and Rosenbauer, 2004).

A number of methods have been developed to determine the level of residual monomer. FTIR (Duray and Gilbert, 1997; Azzarri *et al.*, 2003; Imazato *et al.*, 2001), GC

(Mc Cabe and Basker, 1976; Sadamori and Kotani, 1992; Austin and Basker, 1980) and HPLC (Shim and Watts, 1999; Rose *et al.*, 2000; Urban *et al.*, 2007) have been most commonly repeated.

The amount of residual monomer in the cured resin was dependent not only on the processing method (Jagger, 1978; Miettinen and Vallittu, 1997; Dogan *et al.*, 1995; Wong *et al.*, 1999; Harrison and Huggett, 1992; Kalipcilar *et al.*, 1991; Azzarri *et al.*, 2003; Huggett *et al.*, 1984), but also, on the duration of water immersion of dentures (Tsuchiya *et al.*, 1994; Vallittu *et al.*, 1995, 1998; Shim and Watts, 1999), sample thickness (Fletcher *et al.*, 1983; Huggett *et al.*, 1984), drill speed (Fletcher *et al.*, 1983), surface condition of dentures (Pfeiffer and Rosenbauer, 2004; Vallittu, 1996), usage period of dentures and measuring method (Rose *et al.*, 2000; Urban *et al.*, 2007; Duray and Gilbert, 1997; Azzarri *et al.*, 2003; Sadamori and Kotani, 1992; Austin and Basker, 1980; Imazato *et al.*, 2001).

Numerous investigators have evaluated different methods for decreasing the residual monomer content of acrylic samples. Dogan *et al.* (1995) studied the effect of varying polymerization times and temperature on the residual monomer content of denture base materials. The authors showed that increased temperatures and extended polymerization times were accompanied by decrease in the residual monomer content. Austin and Basker (1980) reported that specimens produced by a short curing cycle contained up to 7 times the residual monomer content of specimens cured by a long cycle. Faltermeier *et al.* (2007) reported that heat as a second curing method, improve the acrylic resins properties.

Tsuchiya *et al.* (1994) demonstrated that the residual monomer content of denture base materials is lowered to a quarter of the initial value if the denture is immersed in water at 50°C for 1 h after polymerization. Vallittu *et al.* (1995) obtained similar results. If the specimens were immersed in water at 37°C for 24 h, considerably more monomer was removed than during immersion in water at 22°C. Accordingly, Vallittu *et al.* (1995) recommended the water immersion of denture in water at 37°C for one day prior to insertion. Shim and Watts (1999) examined the residual monomer content after additional immersion cycles of different durations at 100°C and demonstrated that the residual monomer content could be reduced considerably.

Austin and Basker (1980) suggested that thin plates tend to have a higher level of residual monomer than the thick plate for any one of the curing cycles. This accord with an earlier finding (Huggett *et al.*, 1984) that different part of test dentures had different monomer levels

depending on the thickness of the plate in that area. This effect is probably attributed to the heat evolved during the polymerization reaction. During the curing process this self-generated heat would cause the thicker section to reach higher temperatures resulting in a greater degree of polymerization and a corresponding reduction in the amount of unconsumed monomer.

Fletcher *et al.* (1983) found that high speed drilling may generate heat, which would result in depolymerization of the acrylic resin and may distort the actual residual monomer concentration.

The surface condition of a denture also plays a significant role in the release of residual monomer (Pfeiffer and Rosenbauer, 2004). Vallittu (1996) examined whether a polishing process or a light-polymerized varnish affected the residual monomer content in a denture. The author demonstrated that monomer release reached the lowest level during 2 day soaking of varnished specimens. High-Luster polished specimens, however, revealed clearly lower monomer release than untreated specimens.

Sadamori and Kotani (1992) reported that residual monomer content in acrylic dentures could be detected for up to several years after use. While, it appeared that most of the residual monomer was lost after about 5 years, complete loss of the residual monomer content may take many more years. Hence, it was possible that acrylic dentures may release MMA for long periods of time, but, the level of leachable substances was difficult to detect.

In this study, sample thickness, polymerization method, surface condition, drill speed, storage condition and the measuring method were the same for all of the samples, so these parameters did not have affect the residual monomer content of the samples.

According to ISO 1567 standard, the residual monomer content of denture base materials should not exceed 2.2% wt (Pfeiffer and Rosenbauer, 2004).

Based on the results of the analytical method used in present study, Acropars acrylic resin did not containing any detectable residual monomer and complied the requirements of ISO 1567 regarding residual monomer.

There are numerous studies concerning the amount of residual monomer of various acrylic resins. Austin and Basker (1980) reported residual monomer content for heat cured acrylic resin with longer curing period to be 0.29%, Shim and Watts (1999) results ranging from 0.2-0.4% (Mc Cabe and Basker, 1976) results are from 0.04-0.18% and Sadamori and Kotani (1992) results are from 0.12-4.22%. Huggett *et al.* (1984) have shown that residual monomer in levels exceeding 0.3% is released from the denture base. Craig *et al.* (2000) and Craig (2002) result is

less than 1%. Pfeiffer and Rosenbauer (2004) reported residual monomer content of 2 commercial heat cured acrylic resins to be 0.31 and 0.44%. The results of Vallittu *et al.* (1998) and his coworkers indicate that the amount of residual monomer is 0.07%.

The results of Huggett *et al.* (1984), Shim and Watts (1999), Craig *et al.* (2000), Craig (2002) and Pfeiffer and Rosenbauer (2004) studies are in agreement with the results of the current study for the Acropars samples.

Regarding the importance of residual monomer content, it is suggested to prepare the samples as dentures and to measure the amount of residual monomer in different usage period of these dentures.

CONCLUSION

The residual monomer concentration of Acropars samples was outside the detection limits of the gas chromatography method used in this study, hence this was reported as <0.02%.

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