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# Effect on Working Time of Heat Cure Poly (Methyl Methacrylate) Denture Base Resins at Different Refrigeration Storage Temperatures And Time Intervals - An Invitro Study.

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## Abstract

**Background and Objectives:** One of the greatest disadvantages of heat cure Poly(methyl methacrylate) resin material is its short working time. Therefore, it is desirable to have long working time for manipulation of acrylic resin, particularly during dough stage. It was hypothesized that refrigeration /freezing the mixed dough prior to packing can extend the working time, without adversely affecting properties of polymerized material. Hence, this invitro study was planned to evaluate the effect of refrigeration on working time of heat cure acrylic resin denture base material, when its polymer and monomer dough mix was stored at different temperatures for different time intervals.

**Methods:** Heat cure acrylic resin was mixed and at dough stage, transferred to deep freezing and normal freezing sections of a refrigerator and frozen for periods of 1 day, 1 week, and 1 month. At the end of the storage times, sufficient dough was removed, thawed and tests were done to compare and evaluate the working time before and after refrigeration.

**Results:** showed that working time of the material increased on storing the heat cure PMMA resin dough at two different temperatures in refrigerator. The working time improved significantly after 1 month's storage in deep freezer section of the refrigerator as compared to other storage temperatures and time intervals

**Interpretation and Conclusion**: It is concluded that the heat cure denture base resin at dough stage can be stored in deep freezing section of refrigerator up to 1month with a statistically significant increase in working time. But at the same time, the storage of material in normal freezing section of refrigerator up to 1 week showed no statistically significant effect on the working time of heat cure acrylic denture base material.

**Keywords:** Invitro; heat cure Poly (methyl methacrylate) denture base resin; refrigeration; working time.

## Introduction

Acrylic resin, namely Poly(methyl methacrylate) has been successfully used for various applications in dentistry, since its introduction by Dr. Walter Wright in 1937.[1] It continues to be the material of choice for denture fabrication because of its good working characteristics, ease of processing with minimum equipment and expense, accuracy of fit, stability in oral environment and superior esthetics. According to a study in UK, virtually all dentures constructed in NHS (National Health Service, UK) are made from this material.[2]

Despite its popularity, not all the characteristics of Poly (methyl methacrylate) resin are ideal for use as denture base material.[3]One major disadvantage of Poly (methyl methacrylate) resins is their short working / manipulation time.

Working time may be defined as the time that a mix of denture base material remains in dough like stage.[4] It can also be defined as the period between doughing and setting time in which it is possible to pack the dough.[5] ANSI/ADA specification no.12 requires the dough to remain moldable for at least five minutes.[4] Working time plays an important role in compression molding technique,[4] in which PMMA powder and Methyl methacrylate liquid are mixed to form a plastic mass, which is then forced to flow into mold space, and confine to the shape of the mold. When the powder and liquid are mixed together, the mass passes through a series of stages: sandy, stringy, dough, rubbery and stiff. The material is ready for manipulation at the dough stage. Therefore the material should be packed during the dough stage, since manipulating material too early or beyond this time, results in either breakdown of the material structure or damage to the mold, and consequently influences the accuracy and quality of the molded denture. Ideally, the dough stage should remain in workable condition as long as possible and the dough should exhibit good flow characteristics, so that optimum packing plasticity is achieved.[5]

The flow properties of the acrylic resin depend on factors such as the type and structure of the polymer, concentration of the polymer/monomer mix and technique.[6] Another factor which may influence the flow of the material is variation in temperature [7] during dough-forming and storage of the mix.

The viscosity-limited working time can pose problems, when making a packing involving multiple denture preparations. It may therefore be desirable to extend the working time of Poly (methyl methacrylate) denture base resin when making a multiple packing. Freezing the mixed dough prior to packing can extend the working time.[4] This allows the laboratory personnel to use portions as and when necessary for economy of both material and time supposedly without adversely affecting properties of the polymerized material and it was hypothesized that freezing may also increase the interpenetrating polymer network and hence improve the properties of the polymerized resin.[3]

Hence, this invitro study was planned to evaluate the effect of refrigeration on working time heat cure acrylic resin, when it is stored at dough stage in different temperatures for different intervals.

## Objective

To evaluate the effect of two refrigeration temperatures on the working time of heat cure acrylic resin dough, when stored for different time intervals.

## Methodology

The methodology used in this study has been described in the following order:

1. Mixing of polymer and monomer of heat cure acrylic resin and storing this mixture at dough stage in a

refrigerator, at different temperatures for different time intervals.

2. Evaluation of Working time

Mixing of polymer and monomer of heat cure acrylic resin and storing the mixture at dough stage in a refrigerator, at different temperatures for different time intervals

Polymer powder and monomer liquid of Poly(methyl methacrylate) denture base resin(DPI, Mumbai, India) were mixed as per the manufacturer's instructions in the ratio of 3:1 by volume. The powder and liquid were dispensed with the help of measuring jars. The mixture was left to stand in a porcelain-mixing jar until it reached the dough stage. For evaluation of dough stage of powder / liquid mixtures, a probing test, [5] as given by ASTM specification F451- 76 was followed.

**Probing test**: In this test, approximately two minutes after the onset of mixing, the mixture will be probed gently with a gloved finger (as shown in photo 5). Visual notice will be taken, as to the formation of fibers between the surface of mix and finger as it leaves the surface. This process of probing will be repeated every minute, until the gloved finger separates cleanly from the surface. The time at which this clear separation of finger from the mix occurs first will be denoted as the beginning of dough stage or dough forming time.

When the mix reached the dough stage, it was transferred into stainless steel containers wrapped with aluminum foil and then stored in airtight plastic containers. These containers are immediately transferred into the deep freezer section and normal freezer sections of the refrigerator separately (as shown in photo 8). The temperature measured at deep freezer was ranging from -16 to -18$^{o}$C and temperature measured at normal cooling section of refrigerator was in the range of 10 to 12$^{o}$C. The temperatures were measured using a special lab thermometer. The material was stored for 1day, 1week and 1month respectively at the above mentioned temperatures and later portions of this material was used for measuring the working time. Care was taken to mix and store the whole material required for testing at once, so as to avoid any discrepancies due to repeated mixing and proportioning of powder and liquid. It was also ensured that there was uninterrupted power supply to the refrigerator, by connecting it to a generator. This prevents any changes occurring in temperatures of refrigerator due to sudden power shut down.

**Evaluation of the Working Times of Material**

Before the evaluation of working time for the sake of convenience the material was divided into different groups as follows;

**Group I** - Control group (Material prepared before storing in the refrigerator)

**Group II** - Material after storing for 1 day in deep freezing section of refrigerator (at -16$^{0}$C to -18$^{0}$C)

**Group III** - Material after storing for 1 week in deep freezing section of refrigerator (at -16$^{0}$C to -18$^{0}$C)

**Group IV** - Material after storing for 1 month in deep freezing section of refrigerator (at -16$^{0}$C to -18$^{0}$C)

**Group V** - Material after storing for 1 day in normal freezing section of refrigerator (at 10$^{0}$C to 12$^{0}$C)

**Group VI** - Material after storing for 1 week in normal freezing section of refrigerator (at 10$^{0}$C to 12$^{0}$C)

**Group VII** - Material after storing for 1 month in normal freezing section of refrigerator (at 10$^{0}$C to 12$^{0}$C)

Working times for manipulation of material during dough stage before and after storage for different time intervals at different refrigeration temperatures was determined by using a Needle penetrometer and a stop clock (as shown in photo 6).

**Needle penetrometer test**[5]: In this method, the working/manipulation time of dough was determined by

calculating time difference between the initial penetration and final penetration of the needle. Needle penetrometer used in this study had a 20 gm plunger load and 1mm diameter penetrating needle, with a measuring gauge to measure the depth of penetration of needle. The mix was then placed in a porcelain jar on the table of penetrometer and the needle was allowed to penetrate into the mix every 5 sec. The initial penetration (up to or < 10mm) occurred at the beginning of dough stage and the final penetration (<3mm) occurred at the end of dough stage or the beginning of rubbery stage. The time taken between initial penetration and final penetration was calculated as the working time of dough.

Portions of material from the already mixed and stored resin at different refrigeration temperatures for different time intervals was taken and working time was calculated in the same way as described above. The working times were calculated for all the groups mentioned above, at a room temperature of $28\pm2^{0}$C. The time periods obtained for each group were compared and evaluated with other groups. Measurement of working time for material in group VII was not possible, as the material was not workable.

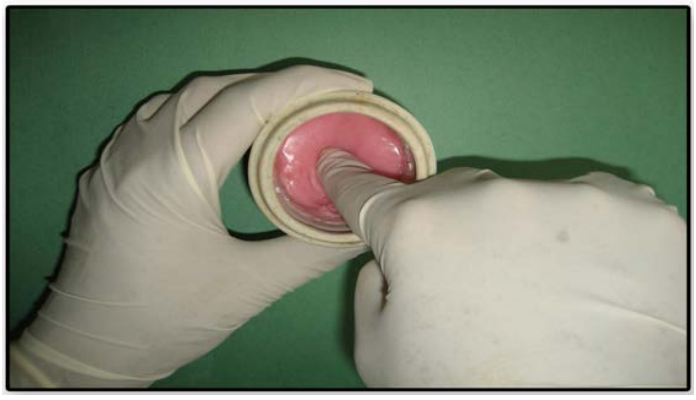


Figure 1 : Probing Test

Figure 2 :Needle penetrometerand digital timer/ stop clock

Figure 3 : Refrigerator used for study


Figure 4 : Storage of PMMA resin at dough stage in deep and normal freezers

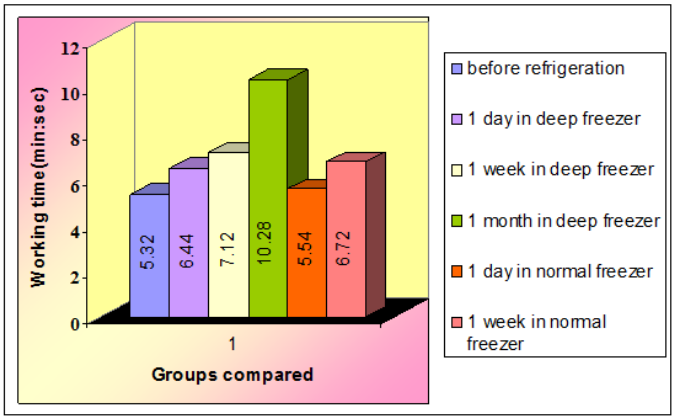
**Results**

The aim of this study is to compare the effect of refrigeration on the working time of heat cure PMMA denture base resin, before and after storing the material at

dough stage in deep and normal freezing temperatures for different time intervals of 1 day, 1 week and 1 month. The results obtained after the study are observed as below:

Table 1: Working times obtained for each study group

| Groups | Description | N | Working time (min:sec) |
|---|---|---|---|
| I | Control group (Material prepared before storing in the refrigerator) | 1 | 05:32 |
| II | Material after storing for 1day in deep freezing section of refrigerator (at -16$^0$C to -18$^0$C) | 1 | 06:44 |
| III | Material after storing for 1week in deep freezing section of refrigerator (at -16$^0$C to -18$^0$C) | 1 | 07:12 |
| IV | Material after storing for 1month in deep freezing section of refrigerator (at -16$^0$C to -18$^0$C) | 1 | 10:28 |
| V | Material after storing for 1 day in normal freezing section of refrigerator ( at 10$^0$C to 12$^0$C) | 1 | 05:54 |
| VI | Material after storing for 1week in normal freezing section of refrigerator ( at 10$^0$C to 12$^0$C) | 1 | 06:72 |
| VII | Material after storing for 1month in normal freezing section of refrigerator ( at 10$^0$C to 12$^0$C) | 1 | Not workable |




Graph1. Comparison of working times (min:sec) of the heat cure acrylic resin material in dough stage before and after refrigeration at different temperatures for different time intervals

## Discussion

Working time for manipulation of material during dough stage before and after storage for different time intervals at different refrigeration temperatures was determined by using a Needle penetrometer and a stop clock, as was explained in the methodology. The results obtained through this method, showed that working time increased significantly from group I to group VI, with maximum working time recorded for material stored in deep freezer section of refrigerator for 1 month (group IV).The lowest value was showed by control group (group I) as shown in table 1 and graph 1. There was also increase of working time for storage of material in normal freezer section of refrigerator for 1 day (group V) and 1 week (group VI),but it was not as significant as group IV. Hence the above findings of this study show that refrigeration of heat cure acrylic resin in its dough stage increased the working time, more significantly when stored at deep freezing temperature up to 1 month.

This phenomenon of increase in working time on freezing can be explained by a study related to effect of temperature on viscosity and working time of acrylic denture base polymers, which stated that as temperature increases the viscosity of the mix increases much more rapidly and manipulation period reduces and when temperature decreases or on cooling the manipulation period increases .[7]

**Limitations of the Study**

The estimation of the working time was not subjected to statistical analysis, as the number of samples tested for each group was only one. So the total number of samples tested was only six, leaving behind the seventh group as the material was not workable. The working time for each sample of material could be measured only once, as it was difficult to replicate the same standardized conditions like mixing the polymer/ monomer ratios, obtaining the exact dough consistency, maintaining the same room temperature, maintaining the same storage conditions every time the test had to be repeated. Hence, the results obtained for the working time evaluation can be considered arbitrary and further studies have to be done to confirm the findings in this study.

Hence the result of this study which shows the increase in working time after refrigerating the acrylic resin dough in deep freezer for a period of 1 month is in accordance with the above mentioned hypothesis. But further studies are required to confirm the findings of this study.

**Conclusion**

Within the limitations of the study, it was concluded that,

- There is economic advantage of deep freezer storage of heat cure acrylic resin polymer/monomer mix during dough stage.
- Refrigerating the heat-cure PMMA resin polymer/monomer mix during the dough stage increases the working time of the material.

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