



## Physical changes in valplast heated at high temperatures as dentures to support forensic identification

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### Abstract

**Background:** Dentures are one of the tools used by forensic experts to determine the identity of an unknown fire victim due to the physical changes at high temperatures. Meanwhile, Valplast is a widely used alternative to metal-based materials due to its physical and mechanical properties such as heat resistance. Therefore, this study aims to analyze the physical changes in shape, weight, and color of Valplast heated at high temperatures.

**Methods:** This experimental study used Valplast heated in a miditherm oven at four temperatures namely 288 °C, 319 °C, 412 °C, and 800°C, while observations were carried out before and after heating using Adobe Photoshop, Digital Scale, and Color Reader.

**Results:** The results showed differences in each group; the higher the heating temperature, the greater the physical changes. Furthermore, the Kruskal Wallis and Mann-Whitney test showed significant differences between the temperatures group with  $p < 0.05$ .

**Conclusions:** Based on the results, physical changes such as discoloration, shape, and weight reduction were observed in Valplast heated at high temperatures.

**Keywords:** forensic identification, valplast, physical change

### Introduction

Fire is a catastrophic event that causes various casualties. Meanwhile, the number of victims requires identification as each has the right to be returned to the family and buried properly according to their respective beliefs [1]. Many disasters result in the significant destruction and fragmentation of human remains, thereby making examination and identification very challenging [2]. Forensic identification of fire victims is essential, not only for humanitarian reasons but also for civil or criminal investigative needs. Identification of unknown individuals is of paramount importance in medico-legal cases to resolve criminal investigations, insurance settlements, proper burial, and grief resolution of family and friends [3]. It is a very challenging task because dead bodies are often mutilated to such an extent that they cannot be identified by general physical examination alone [4].

Identification of human remains in mass disasters is a difficult task. Dental examination for identification is an accurate and easy method given that the teeth are the strongest part of the human body, which can withstand high explosions and are not damaged by such incidents [5]. Teeth are considered to be the most indestructible components of the human body and have the highest resistance to most environmental effects like fire, desiccation, and decomposition because of their particularly resistant composition and protection by soft tissues [3]. A dental examination is carried out by comparing the dental data collected previously from an individual, (antemortem data), with the results of an unknown victim (postmortem data) [6]. The use of forensic odontology frequently plays a major role

in identification after many multi-fatality incidents. As with all applied methods, it is dependent on the availability of adequate antemortem dental records together with the skill and methodology of the postmortem examination of the structures of the dentition and surrounding tissues and finally upon the effective and accurate comparison of these antemortem and postmortem observations [2]. Victims that had previously extracted teeth and wore dentures are identified by observing the degree of exposure to heat. The identification is carried out by observing the color change in the denture base used by the victim before the fire incident [7].

Various kinds of denture models have been traced and identified. There are several choices of base materials, namely ceramics, metals, and other types of polymers. Dentures found in victims are used as a source of post-mortem data. Furthermore, it hastens the identification process and increases accuracy, but is often difficult without complete antemortem dental data [8].

Several studies have explored the physical changes of dental materials exposed to high temperatures ranging from composite resins, amalgam, and Glass Ionomer [9]. However, there is no report on physical changes, namely shape, weight, and color due to high-temperature heating using Valplast. Therefore, this study aims to analyze the physical changes in *shape, weight, and color of Valplast, heated at high temperatures.*

### Materials and Methods

#### Sampling Stage

The printed pattern is made of wax in the form of a block with a size of  $25 \times 15 \times 2.5 \pm 0.1$  mm, while the wax model was molded in a flask with a hardened (setting) gypsum stone

and the entire surface was smeared with vaseline. Furthermore, boiling was carried out to remove wax from the mold by placing the flask in boiling water for 4 to 6 minutes to obtain a mold cavity. The mold was smeared with CMS (Cold Mold Seal) and then filled with Valplast in the form of a cartridge in a furnace heated to a temperature of 288°C (550°F) for 11 minutes. Moreover, a cartridge containing liquid Valplast was placed in the injection unit and then pushed into the flask using a plunger. The Valplast injection was removed from the flask and then polished.

**Treatment Stage**

The Valplast was first weighed using a digital scale and then placed on ceramic plates in each group (P1, P2, P3, P4) before heating in the oven to an initial temperature of 32 °C. Furthermore, the samples in the first group (P1) were placed in an oven and heated up to 288 °C. The sample was then removed and allowed to cool for about 10 seconds. After cooling, the change in color, shape, and weight of the Valplast was observed. Similar procedures were followed for groups P2, P3, and P4.

**Weight Calculation Stage**

The difference between the initial and final weight of Valplast before and after heating was calculated using a digital scale.

**Form Observation Stage**

This was carried out using a superimposition technique, where the photos of the sample before and after heating were captured and then interpreted using the Adobe Photoshop CS6 program. The photo after heating was placed on top and the transparency level (opacity) was lowered. Furthermore, the shape observation was carried out by comparing the sample before and after heating.

**Color Measurement Stage**

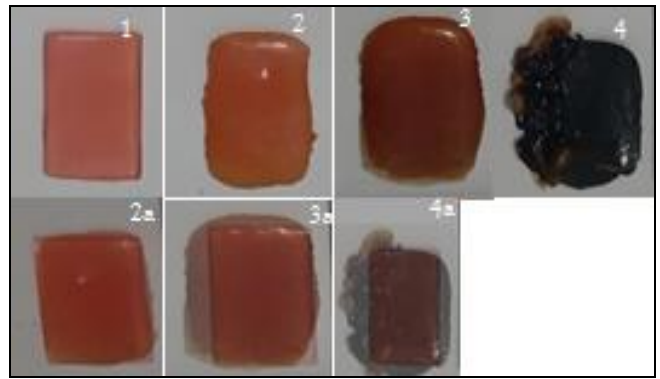
Valplast color measurements were carried out before and after heating under the D65 illumination standard which corresponds to the average daytime. The color changes were determined using a color reader Minolta CR-14.

**Results**

The results of the study as presented in Table 1 show that all samples experienced a change in weight after heating at each temperature. The results of changes in the shape of Valplast after heating using the superimposition method are presented in Figure 1.

**Table 1:** The average difference in weight of Valplast after heating

Groups	N	The average change in weight of Valplast (grams) ± SD
288°C	6	-0,015 ± 0,005
319°C	6	-0,018 ± 0,012
412°C	6	-0,367 ± 0,012
800°C	6	-



**Fig 1:** Changes in the Valplast's Shape. Note: Valplast before heating (1), Valplast after heating at 288 °C (2), heating at 319 °C (3), heating at 412 °C (4), adobe photoshop at 288 °C (2a), adobe photoshop temperature at 288 °C (2a), adobe photoshop temperature at 319 °C (3a), adobe photoshop temperature at 412 °C (4a).

After heating, there was a change in all sample groups. Changes in P1 (288 °C) and P2 (319 °C) had relatively similar patterns as the original, but there was a slight change in the corners, which appeared rounded. Meanwhile, the largest change was observed in P3 (412°C), where the original or initial shape had changed significantly. Furthermore, changes in P4 (800 °C) were not measured because the Valplast was lost from the ceramic crucible during the heating.

Based on Table 2, there was a color change in groups P1, P2, and P3. Meanwhile, the ΔE\*ab value of P4 which was exposed to a temperature of 800°C was not measured as all the Valplast placed in the ceramic dish was lost during the heating process.

**Table 2:** The average value of the color change on Valplast after heating

Group	N	Mean of Valplast color change (ΔE*ab) ± SD
288°C	6	5,008 ± 2,548
319°C	6	22,496 ± 7,895
412°C	6	33,810 ± 3,544
800°C	6	-

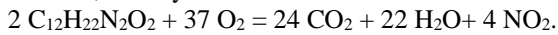
**Discussion**

Identification of a burned subject through dental procedures compares post-mortem records with antemortem dental clinical history. Identity verification of burned victims is hindered by the post-mortem state of the victim and the amount and quality of antemortem data [6]. Teeth and other restorative materials are only exposed to heat from flames but do not catch fire immediately [10]. A dental examination is carried out by observing the condition of the dentures which is considered important in the forensic identification process [11]. Meanwhile, the most commonly used removable partial denture base material is Valplast. After heating at high temperatures, it undergoes physical changes, in weight, color, and shape. These changes are used in the identification process of fire victims [11]. Therefore, this study was conducted to determine changes in weight, color, and shape of Valplast, heated at high temperatures. The tool used to control the temperature was an oven which is analogous to the oral cavity.

Based on the results, there were changes in shape, weight, and color after heating at high temperatures. At 288 °C and 319 °C, there was no significant change but at 412 °C, there

was a significant change in shape, where the surface was uneven and was no longer square. The factor responsible for this deformation is  $T_g$  (glass transition temperature) which prevents softening and damage during heating. A Valplast sample heated above a  $T_g$  of  $130^\circ\text{C}$  formed a new shape in which the molecular motion begins to push the chains apart and soften the polymer [12, 13]. Another factor is that heating produces a thermal decomposition reaction, namely the breakdown of chemical bonds into gaseous form. The thermal decomposition temperature of nylon 6.6 (thermoplastic nylon) ranges from  $80 - 310^\circ\text{C}$ . Furthermore, simple compounds produced from decomposition reactions are similar to the products of combustion reactions. The simple compounds formed might include gaseous products, meanwhile, the decomposition gases identified in the air include  $\text{CO}$ ,  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ ,  $\text{NH}_3$ ,  $\text{HCN}$ , and  $\text{NO}_x$ . Moreover, the thermal decomposition of some nylons is caused by free radical formation and bond cleavage [14]. For example, thermoplastic nylon thermal decomposition leads to the cleavage of the  $\text{NH-CO}$  bond which is detected by mass spectrometry (MS) [14].

The heating of Valplast is an oxidation-reduction (redox) reaction, namely



The higher the heating temperature, the faster the formation of volatile combustion products. Furthermore, complete combustion takes place at  $800^\circ\text{C}$  and is indicated by the formation of  $\text{CO}_2$  and  $\text{H}_2\text{O}$ , therefore, Valplast is lost from the ceramic cup. All Carbon (C) contained in Valplast forms  $\text{CO}_2$  after heating, while all Hydrogen (H) changes into  $\text{H}_2\text{O}$  [15]. This is due to heating at very high temperatures above the melting point [14].

There was a reduction in the weight of the Valplast due to the exposure to higher temperatures as shown in table 1. At  $288^\circ\text{C}$ ,  $319^\circ\text{C}$ , and  $412^\circ\text{C}$ , there was a reduction in weight with an average of 0.015 g, 0.018 g, and 0.367g, respectively. The higher the temperature, the greater the weight reduction. This is due to the evaporation of the samples into gases at higher temperatures. The reduction detected by the mass spectrometer (MS) was approximately 5% of the original weight of the thermoplastic nylon sample. At  $400^\circ\text{C}$ , thermoplastic nylon produces gaseous products namely carbon dioxide and water [14].

The heating process caused color changes in Valplast as shown in table 2. The largest color change was found at  $412^\circ\text{C}$  with a mean of 33,810. Initially, the color was pink but became darker after heating as indicated by the higher  $\Delta E^*$  value. These changes occur due to intrinsic and extrinsic factors. Intrinsic color changes occurred due to chemically changing conditions such as heat [16].

Meanwhile, extrinsic factors such as discoloration occurred due to the absorption of water in the denture base material. Valplast has high water absorption properties, this is because the water molecules penetrating between the molecular chains are caused by hydrophilic amide bonds and form the main chain of the polyamide resin [17]. Water molecules diffuse into the polymer matrix due to the small size, less than 0.28 nm. This is smaller than the polymer chain spacing in the matrix [18]. Moreover, water penetrates the polymer through unsaturated chains or imbalances in the strength of the intermolecular bonds [19].

Discoloration of Valplast after heating is also caused by the presence of microporosity. Most of the samples in this study contained microporosity which was presumably formed

during the injection molding process due to the entry of air during the heating procedure [18]. This condition creates an empty area, which is then filled with water [20], meanwhile, the penetration of water into the Valplast severs the chemical chain [18]. The water molecule,  $\text{H}_2\text{O}$ , has an H atom that penetrates the chain and binds with the C atom in Valplast, therefore the initially long bond, namely  $\text{CH}_2\text{-CH}_2\text{-CH}_2$  is cut into  $\text{CH}_2$  and  $\text{CH}_3\text{-CH}_3$ , consequently, the amount of  $\text{CH}_3$  is higher than  $\text{CH}_2$ . Chain cleavage causes the chemical bonds to become irregular and stretches the intermolecular space. Furthermore, the initially brownish-yellow Valplast becomes black with increasing temperature at  $412^\circ\text{C}$ . This color change is probably due to the degradation in the form of polymer breakdown in diamine and dicarboxylic acid caused by heating and oxidation, as well as carbonization in the Valplast pigment. In the oxidation process, oxygen binds to hydrogen atoms and evaporates with increasing temperature, therefore only carbon atoms are left which form hexagonal groups. This process is called carbonization which turns the color black [21, 22].

Dentures facilitate accurate and fast identification of fire victims but the process is difficult without complete antemortem dental data [8].

Meanwhile, physical changes in Valplast tend to be different with direct fire exposure. Based on the results, changes in color, weight, and shape of Valplast tend to increase with the intensity of the fire temperature on the victim. These results are expected to be used as support for the forensic identification process.

## Conclusion

In conclusion, in our study Valplast undergoes a change in color, shape, and weight loss after being heated to high temperatures. Valplast weight decreases more and more as the temperature rises. The shape of the Valplast changes when exposed to high temperatures, and at a temperature of  $800^\circ\text{C}$  the shape and weight of the Valplast cannot be detected because it has become ash.

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## Conflicts of interest

The authors declare that there is no conflict of interest.

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