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A **New Micro-composite Material of Micro-particle Amalgam/Polyvinyl Alcohol for Teething Structures**

Falak O. Abas*[1], Raghad U. Abass[2]
[1]Chemical Engineering Department, University of Technology, Baghdad, Iraq
[2] Applied Science Department, University of Technology, Baghdad, Iraq

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Abstract

Dental amalgam is a mixture of approximately 50% mercury and varying ratios of silver, tin, zinc, and copper. Dental amalgam is a major source of mercury pollution because it is readily absorbed through 90-100% vapour and the oral mucosa. In addition, in certain situations with the oral environment, various types of metallic orthodontic brackets are highly aggressive and can lead to corrosion. However, polyvinyl alcohol (PVA) material has no cytogenetic effects on human health or the environment and is therefore applied in the manufacturing of the new composite material. Different additives from the bonding agent (PVA; 2.4, 4.8, and 7.2 g) dissolved in about 10 ml of water, heated on a hot plate under a high-temperature heat treatment (150-200 ^{O}C), and continuously stirred for about 20 minutes, until all the PVA dissolved or the solution became colorless. Subsequently, a fixed amount of powdered amalgam (2.4g) added after the mercury content was separated. It was continuously mixed until it reached a homogenous solution, then molded and cured to give the final product. New samples were used for the following purposes: (1) to investigate their effects on the chemical, thermal and mechanical properties of the composite samples, (2) composite scanning and images from the EDS diffraction. Scanning lectron microscopy (SEM) indicated that the presence of the methyl group (CHI) that lowers the crystallinity of PVA, also forms bridges between the different chains obtained to indicate the suitable materials for guided dental applications.

Keywords: micro-amalgam, polyvinyl alcohol gel, new composite, dentalfilling, supports structures.

مادة جديدة مايكروية من ملغم دقائق مايكروية/ كحول متعدد الفينييل لهياكل الاسنان

فالك عباس*[1]، رغد عباس[2]
[1]قسم الهندسة الكيميائية ، الجامعة التكنولوجية ، بغداد ، العراق
[2]قسم العلوم التطبيقية ، الجامعة التكنولوجية ، بغداد ، العراق

الخلاصة

ملغم الأسنان عبارة عن مزيج من حوالي 50٪ من الزئبق، إلى جانب نسب مختلفة من الفضة والقصدير والزنك والنحاس. و يعتبر ملغم الأسنان مصدرًا رئيسيًا لتلوث الزئبق لأنه يمتص بسهولة من خلال بخار الفم (90–100٪) ومن خلال الغشاء المخاطي للفم. بالإضافة إلى ذلك، وفي حالات معينة من البيئة الفموية، تكون الأنواع المختلفة من الأقواس المعدنية لتقويم الأسنان شديدة التآثر ويمكن أن تؤدي إلى التآكل. ومع ذلك،

*Email: falak_usama@yahoo.com

فإن مادة polyvinyl alcohol (PVA) ليس لها أي آثار وراثية خلوية على صحة الإنسان أو البيئة، وبالتالي يتم استخدامها في تصنيع المواد المركبة الجديدة. حيث يتم إذابة إضافات مختلفة من عامل الترابط (PVA) مثل (2.4 ، 4.8 ، و 7.2 غم) في حوالي (10 مل) من الماء، ويتم تسخينها على صفيحة ساخنة تحت معالجة حرارية عالية (150−200 درجة مئوية)، ويتم تحريكها بشكل مستمر لمدة 20 دقيقة تقريبًا، حتى يتم إذابة كل PVA و يصبح المحلول عديم اللون. بعد ذلك، تتم إضافة كمية ثابتة من ملغم المسحوق (2.4 غم) بعد فصل محتوى الزئبق. يتم خلطها باستمرار حتى تصل إلى محلول متجانس، ثم يتم تشكيلها ومعالجتها لإعطاء المنتج النهائي. و قد تم تطوير عينات جديدة من خلال: (1) دراسة آثارها على الخواص المميزة− الكيميائية والحرارية والميكانيكية للعينات المركبة. (2) الدراسة من خلال صور مسح الحيود الالكتروني EDS والمسح الإلكتروني المجهري (SEM) التي أشارت إلى أن وجود مجموعة المثيل (CHI) التي تقلل من تبلور PVA، و تشكل جسوراً بين السلاسل المختلفة التي يتم الحصول عليها إشارة إلى كونها مواد ملائمة لتطبيقات طب الأسنان.

Introduction

Posterior composite restorations are common substitutes for mercury-containing silver amalgam restorations because they are mercury-free and less expensive than cast metal. Their longevity is controversial because of the many variables in the oral environment, such as temperature shifts and chemical changes. The main difference between composite and metallic restorations is that composites are water phillic, whereas metallic ones are not. The resin may swell or contract, thereby contributing to the possible failure of the restoration. Additionally, the environmental impact assessment recognized the concerns with regards to the release of mercury from dental amalgams [1, 2]. The exact amount of mercury released by each dentist is difficult to determine, but several studies were conducted over the past ten years and, in general, each dentist in those studies released an average of 56 to 270 mg of mercury per day into the wastewater stream.

The immunological and cytogenetic effects for polyvinyl alcohol (PVA) were studied by Azhar H. and Falak O. Abas [1], who concluded that there were no cytogenetic effects on human and animals, in addition to the high biocompatibility and biotribological properties for this material [2,3]. Polyvinyl alcohol gels (PVOH) are applied as articular repairing materials due to the high adhesion, durability performance [4] and porous free water group's structures [5]. However, nano-structure gels are used in medical applications due to their bioactive properties [6] as interfacial bonding agents for natural tissue to help improve the strong bonding and mechanical contact between the base material and natural fibre [7]. Nano hydroxyapatite-reinforced polyvinyl alcohol (nano-HA/PVA) gel composites were set by mixing nano-HA particles reformed by a silicon combination agent with physical saline solution (PSS) of PVA while approving the freezing-thawing method. The effects of various features and the content of nano-HA subdivisions on the mechanical activities were both studied. Composites are hardened with the introduction of an initiator (usually a light) into an already-existing paste formulation. However, a composite that appears hard does not necessarily mean that it is completely cured, therefore, the curing of composites was achieved with heat to provide a deeper and more infinite type of cure. It is theorized that tempering the composite causes an increase in polymerization, which in turn causes an increase in tensile stress within the composite. Mouthwashes may vary in their chemical composition. Some contain alcohol, which can reach up to 27% in volume, while others contain none [8, 9].

Polymer composites are mostly applied in dental restorations according to their filling ability [10, 11]. Several types of research were achieved to investigate synthesis and characterization of different composite materials dimethacrylates containing quaternary ammonium and methacryloyloxy dodecyl pyridinium bromide (MDPB) functionalities for dental applications antibacterial resin composite to improve dental restoratives also uses as tissues in clinical performance [10-16], or applied as carrier restoration margin in a different green composite system for nano silver material [12]. Other authors tried to synthesize and characterize antibacterial dental monomers and composites, such as amorphous calcium phosphate dental nano-composite and TiNi orthodontic, as ultra morphological studies on the improvement of fluoride-releasing dental bonding agent involving new pre-reacted glass ionomer (PRG) fillers and surface modification arch wires [13-15]. New antibacterial biofilm dentin primers were applied with quaternary ammonium and silver nanoparticles containing amorphous calcium

phosphate nanoparticles for caries [16-17]. Lately, CaP nanoparticles were manufactured via a spray-drying procedure [18]. The new nanocomposites comprise nanoparticles of unstructured calcium phosphate (NACP), free calcium ,and phosphor ions, like to modern CaP composites [19-20]. These can be combined into the antibacterial bonding agent without cooperating the dentin bond power. It is also essential to progress caries-inhibiting adhesives – adhesive bonds of the composite to the tooth structure and infiltrate to the pure infections root teeth [21]. Strong nanocomposites with free radicals as Ca, PO_4, TiF_4 and free flour F release for carring inhibition Long-term antimicrobial polyamide 6/silver-nanocomposites applied around orthodontic brackets [22-23]. The effects of titanium tetrafluoride (TiF_4) and PO_4 application around orthodontic brackets. Nanoparticles of these ions improved the mechanical application properties [24]. In addition, the new fluoride (F) ions improved the glass ionomer [25]. The application of titanium tetrafluoride (TiF_4), chlorhexidine, and fluoride ions around orthodontic brackets improved their aging and mechanical properties [26, 27]. However, dimethyl-acrylate monomers that contained ammonium groups were modified as antimicrobial materials that reduced the bacterial colonization. Therefore, the antibacterial bonding agent was improved using MDPB and other antiseptic agents [28]. It is also beneficial to render the primer antibacterial because it comes directly in contact with the tooth structure [29], application another composite system from quaternary ammonium dimethyl-acrylate (QADM) reinforced with nano silver NAg material for antibacterial applications. The validation was that, while QADM and NAg could remove remaining bacteria in the tooth crack and dentinal tubules, recently applied chlorhexidine particles were used as fillers in dental composite systems to reduce the growth of bacteria [30-31]. The hydrocarbon groups CH_X (x; the hydrogen number) were combined with glass ionomer to give an active antibacterial fluoride system [32-33].

The present work aimed to develop an antibacterial composite micro-particle material (MAg) , improve the capabilities of chemical resistance under different tooth restoration conditions by the use of bonding agents such as PVOH, and characterize the final physical, chemical, and mechanical properties of ther final composite micro-particle material. Experimental

Materials and Methods

Materials

Silver amalgam micro-particles (AgMP): A representative silver–mercury amalgam powder (BTU Company) commonly used in medical teeth applications and has a pore size of less than 65 μm (microns).

Bonding agent polyvinyl alcohol (PVA): An extremely pure (molecular weight 13–23 kDa, grade of hydrolysis 87–89%) compound of PVA (BTU Company) .

Methods

Preparation of Highly Concentrated Stable Dispersions of Uniform Silver Micro-particle (AgMP)

The preparation of highly concentrated stable dispersions of uniform **AgMP** was achieved through the transfer of an aqueous silver nitrate solution (80 ml aqueous) containing 5 wt % of $AgNO_3$ to ascorbic acid solution (17 ml; 1.5 M) at an organized flow rate (2.5 ml /min). The initial pH was lowered by adding perchloric acid to the silver nitrate solution. After that, the silver preparations were washed with deionized water to near neutral pH and redispersed in water to avoid forming dry precipitate. Next, the components were separated, washed with acetone, and dried in a vacuum at a low temperature of 37^{O}C. The dry silver elements were redispersed in deionized water in an ultrasonic soak to obtain intense dispersals. Lastly, the micro-sized silver was transformed into a dried precipitate by freeze dehydration [15].

Preparation of Bonding Agents

PVA used as a bonding agent resin with amalgam for the fabrication of composite materials. PVA first dissolved in 10% vol. of water under high temperatures (150-200 ^{O}C) with constant stirring (500 rpm). Once the PVA dissolved, the aqueous solution became very viscous. Different additive contents of binding agent PVA (2.4, 4.8, and 7.2 g) used in the formulation of amalgam/ PVA composite material. The bonding agents (PVA) reported to secure the silver micro-particle AgMP and develop adhesion to the solid porous support surface [9].

Preparation of Dental Amalgam Composite

Base micro-particle silver AgMP receives a (PVA) reins bonding agent that forms a dimensional network, whereby amalgam/ PVA are loaded onto the polymer network. About 2.4g of polyvinyl alcohol (PVA) was dissolved in about 10 ml of water and heated on a hot plate under high-temperature (150-200 ^{O}C) and continuously stirred for about 20 minutes until all the PVA was dissolved or the solution became colorless. About 2.4g of silver micro-particle AgMP was dissolved in about 10 ml water and stirred continuously for about 20 minutes until all the silver micro-particle AgMP was in a suspended state. Subsequently, both materials were mixed in a beaker and constantly stirred (500 rpm) until all the contents formed a uniform paste. To fabricate this amalgam / PVA composite material, a small amount of Tung oil was added and the paste (AgMP/PVA) was transferred to a mold and left to dry for about 24 hours. Table -1 demonstrates the designed additives of amalgam/ PVA composite material [34].

Table 1-The designed compositions for amalgam/ PVA composite material

Amalgam base (Ag MP) **g**	**2.4**	**2.4**	**2.4**	**2.4**	**0**
PVA bonding agent (g)	**0**	**2.4**	**4.8**	**7.2**	**2.4**
Mixing ratio amalgam/ PVA	**100 / 0**	**50/50**	**67/33**	**75/25**	**100/0**

Analysis of Materials

A scanning electron microscopy (SEM) image of nanoparticle amalgam/ PVA composite material was produced before and after chemical treatment. The SEM used herein is a JEOL JEM-ARM200CF, Germany, with a cold field emission source; CEOS probe aberration corrector, Gatan digital cameras, Oxford Energy Dispersive. Also, EDS (Quantax, Germany) was used to perform the elemental scanning for samples before and after the modification process, using the same auxiliary used for the SEM scan.

In addition, chemical resistance of dental caries restorations materials to the oral environment was characterized at different pH levels (6.2, 7, 7.5) for 0, 24, 48, 72, 96 and 120 hours. Thermal conductivity by Lee- disk and a mechanical hardness test were achieved for the composite system (amalgam/ PVA).

Thermal conductivity was tested by using the Lee- disk (kocyigit Electron DC 0-30 volt and eh 6 Am, USA instrument) for prepared samples of 1cm thickness and 3cm diameter under the conditions of the lab (30ºC). The sample was introduced between two disks made of copper which were then heated. The change in temperature for the sample and the two disks until the falling down of the sample was then used by the following equations to calculate the thermal conductivity K:

$$e = P/\pi r\,[r\,(T_1 + T_3) + 2\,(d_1T_1 + 0.5ds\,(T_1+T_2) + d_2T_2 + d_3T_3] \quad \text{…..(1)}$$

$$K = eds\,[T_1+2T_1\,(d_1 + 0.5ds)/r + T_2ds/\,r] / (T_2 - T_1) \quad \text{……..... (2)}$$

Where:

e = loss in heat per unit area in (w /cm^2.ºC).

P = supplied power in (w).

r = radius of disk in (cm).

d_1, d_2, d_3 = thickness of disks in (cm).

ds = thickness of specimen in (cm).

T_1, T_2, T_3 = measured temperatures of disks no., 1, 2, and 3 in (ºC).

K = thermal conductivity in (w / cm. ºC).

Chemical properties: This type of test was determined by immersion of both base and prepared composite systems in artificial saliva with different pH levels (6.2, 7, 7.5) for 0, 24, 48, 72 and 96 hours at 30□C. The change in weight was recorded every 24 hours to calculate the moisture percentage, absorbency values, and chemical resistance in each sample, and to check which of the samples was more chemically stable than the others (optimum one). For these purposes, the following formula was used:

$$\text{Moisture (\%)} = [(Wi - Wd)/\,Wi] \times 100 \quad \text{-------- (2)}$$

Where: Wi, and Wd estimate the weight for samples initially and after immersion in distilled water in (g).

Hardness property: Hardness is an important property which measures tooth brackets and their ability to withstand deformation by indentation or scraping. Durometer hardness by shore D (J) also serves as a masticatory surface on which food is crushed, ground, and chewed. Specifically, measuring tooth microhardness is a valuable process not only in assessing its plastic properties in relation to masticatory forces but also as a consideration in the selection of restorative materials. Thus, in addition to the properties discussed above, the ability of a tooth to withstand masticatory forces may be related to its mechanical properties [13].

Results & Discussion

Figure-1 shows the base materials, the manufacturing products (amalgam/ PVA composite materials), and their appearance before and after the above described preparation procedure.

Figure 1-Pure and composite amalgam/ PVA composite.

Figure-2 shows the effects of the bonding agent additive PVA (wt.) on the mechanical hardness property of amalgam/ PVA composite material upon application during the manufacture of brackets. We observed that the hardness property of the products amalgam/ PVA composite materials was improved as compared to that of the base material (pure amalgam, MAg). We suspect this improvement was achieved due to the high molecular weight of the bonding agent (PVA). Chemical compatibility of the manufactured material was achieved by the treatment conditions, i.e., by heating and then applying cold pressing techniques.

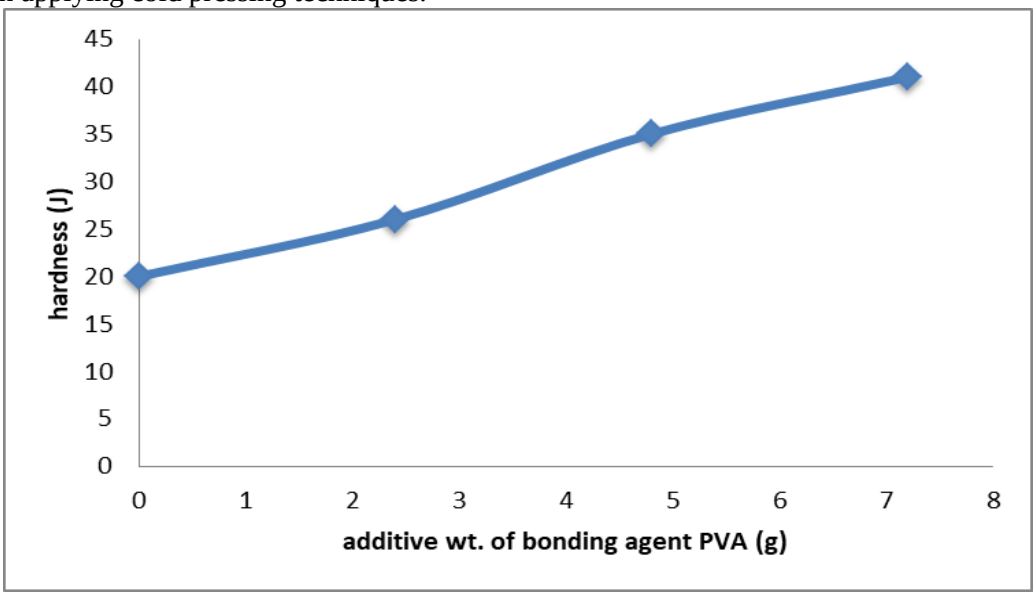

Figure 2-Effect of bonding agent PVA (wt) on the hardness property

Figure-3 shows the effect of the application of the bonding agent additive PVA (wt) on the thermal conductivity properties of the amalgam/ PVA composite material during the manufacture of the brackets; the high molecular weight of PVA induced the formation of a high amount of polar OH groups, which were found in the manufactured material. Also, a high thermal stability (at temperatures between 200–300°C) was conferred upon application of the amalgam/ PVA composite material, whereas the melting point of the base material amalgam was 50°C. The largest improvement in thermal stability was observed with sample 3, which consisted of a 67: 33 (weight: weight ratio) of PVA: MAg.

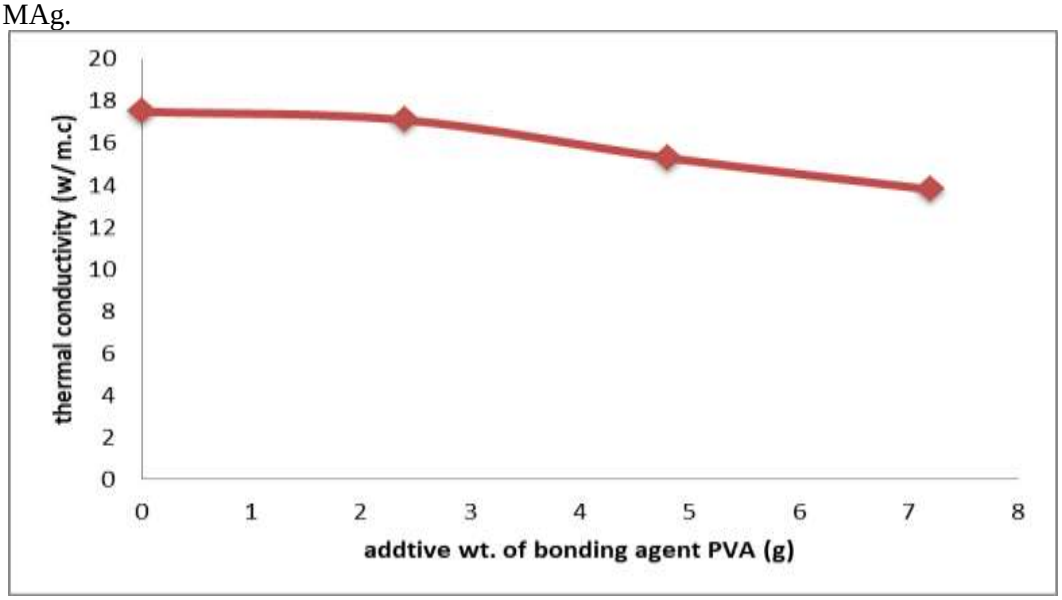

Figure 3-Effect of bonding agent PVA (wt) on the thermal conductivity property

Figures-4, 5, and 6 show the effects of the application of the bonding additive PVA (wt) during the manufacturing process of amalgam/ PVA composite material on the chemical activity (w/w % H_2O) of the brackets, under different acidity conditions (pH= 6.2, 7, and 9) and after different time intervals (after 0, 24, 48, 72, 96, and 120 hrs). Sample three (a weight:weight ratio of 67:33 of PVA: MAg) revealed the highest chemical resistance (i.e., 0.1–0.5%) to an artificial saliva solution (of pH = 7 or pH = 8), due to the large amount of hydroxyl groups which confer chemical stability upon the manufactured material. Pure amalgam showed a low chemical resistance (0–0.01%) to the acidic solution (pH= 6.2); other solutions reached 0–45%, while the optimal sample reached 20%.

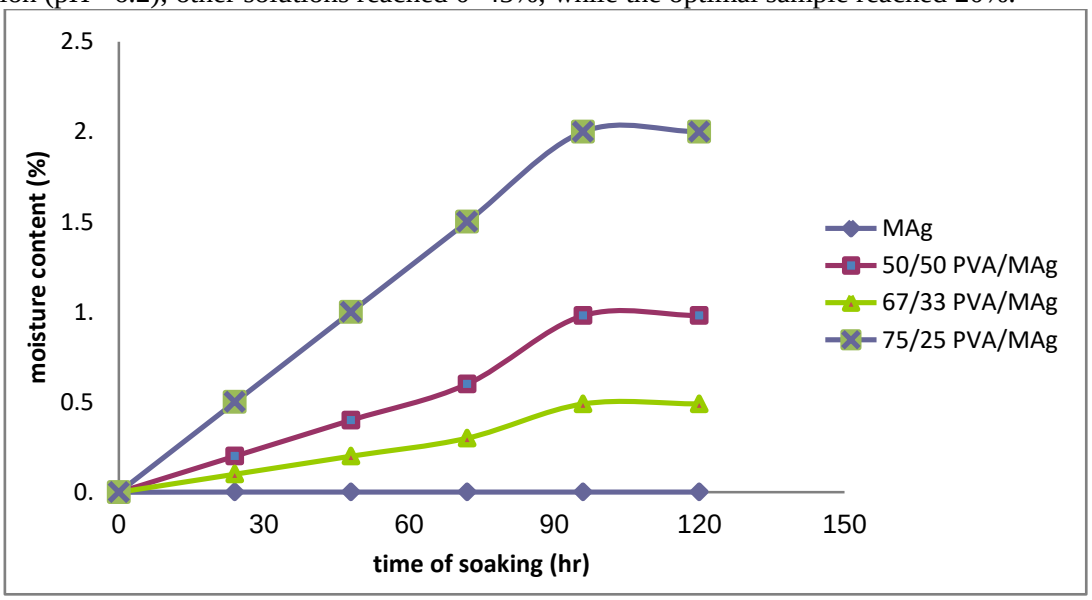

Figure 4-Effect of bonding agent PVA (wt) on the chemical activity property under natural acidity solution (pH 7).

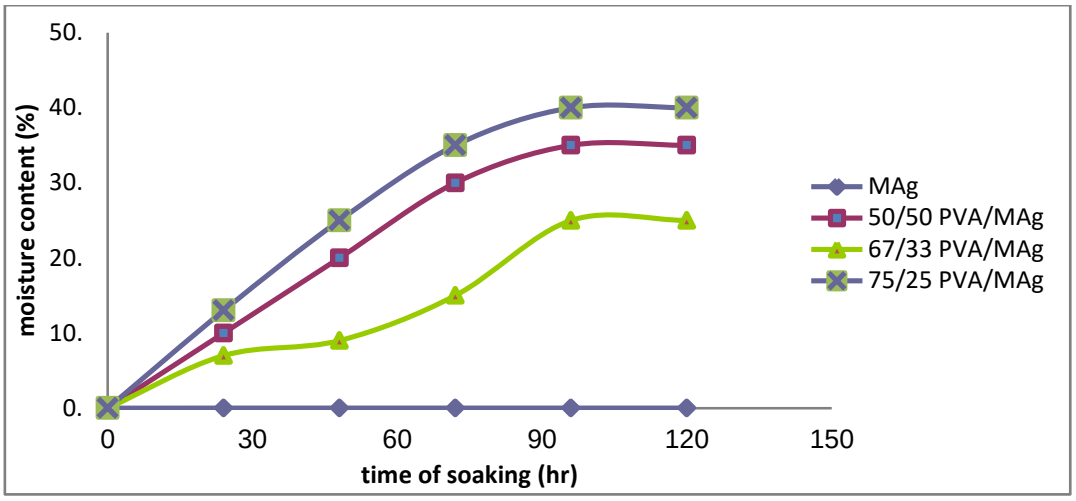

Figure 5-Effect of bonding agent PVA (wt) on the chemical activity property under acidic acidity solution (pH 6.2).

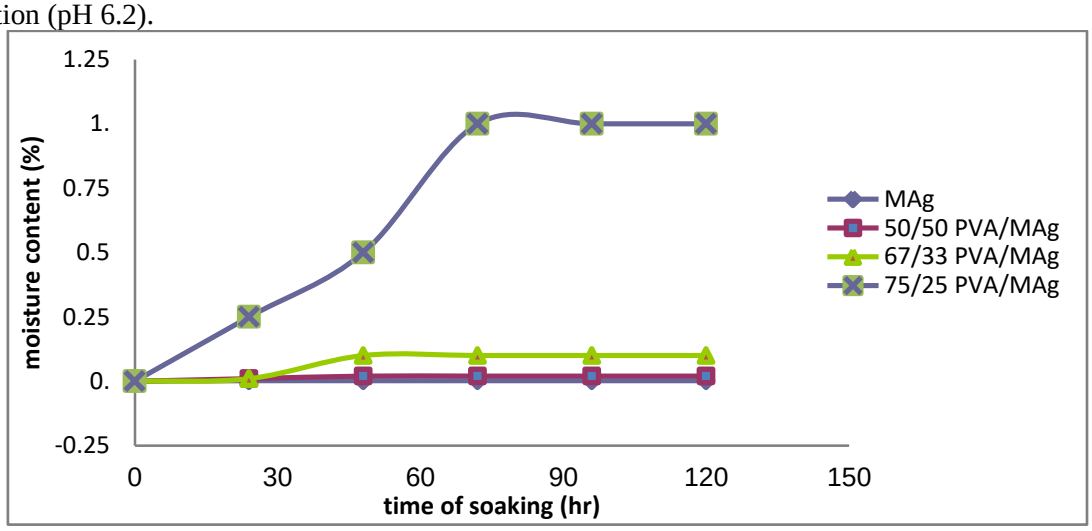

Figure 6-Effect of bonding agent PVA (wt) on the chemical activity property under alkali acidity solution (pH 7.5).

The effect of artificial saliva and rapid changes on the surface of the material was tested using the SEM microscope for the optimal silver amalgam / PVA mix, as shown in Figure-7 (a, b, c, d, e and f). One of the results obtained was that the solutions based on artificial saliva solution had corrosion effects due to the presence of chloride ions, as well as the effect of the change in acid function (8, 7, 6). The presence of chloride ions in the saliva solution stimulates and causes corrosion. Using SEM to study the surface of the overlapping material revealed results and general appearance of the surface of the overlapping substance (PVA), immersed in artificial saliva (Figure-7a). We observed that the images produced by the microscope indicated that the original surface of the ideal superconductor created a high porosity network and was an integral part of the overlapping PVA. As for Figure-7 c and d, SEM revealed the formation and composition of many effective resin bonds and aggregates within the ivory tubes. The resulting images showed that the film layer was relatively high and metabolic, and that the continuous addition of the PVA bonding factor to the micronutrient silica material for silver was effective in reducing the formation of the film-vital layer, in addition to minimizing the production of lactic acid. The results also indicated that the initial use of PVA antibiotic bonding has anti-bronchial effects. This powerful and effective ingredients are considered as primary and essential antibacterial agents that can be very useful in applications, such as repairing the spaces between the teeth which cause damages due to direct contact with the toothpaste. This effect also reduces the bacteria that seep into the ivory tubes and is then ready to clear the dental cavity and eliminate the remaining bacteria. As shown in Figure-7 e, f, the examination using the SEM showed that many voids were formed as a result of corrosion. The formation of the blanks is inevitable due to

the chemical interaction between the metal and the chemical solutions that are put into or produced inside the mouth.

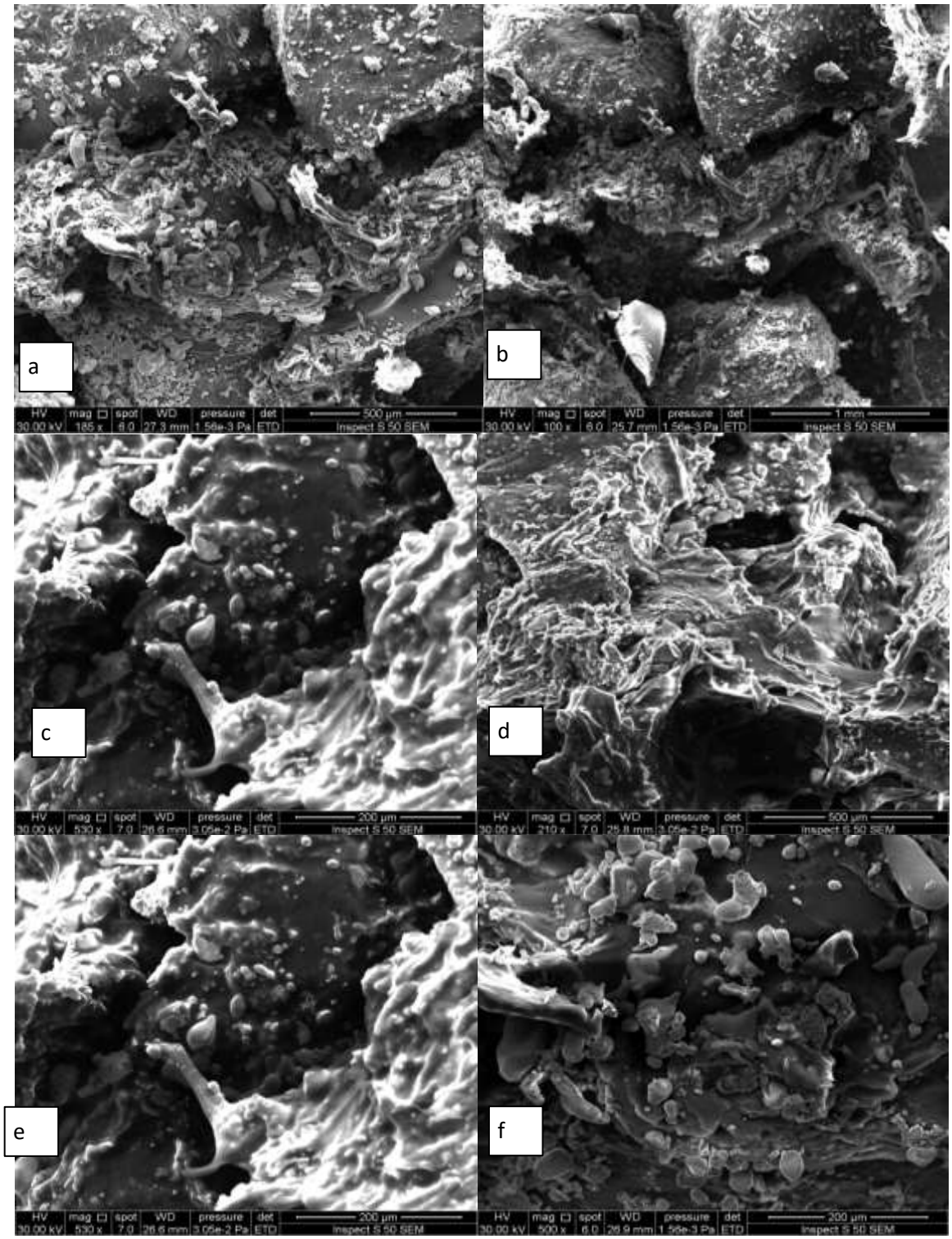

Figure 7-(a, b, c, d, e and f) Surface study of the optimum mixture 67/ 33 amalgam MAg/ PVOH gel before and after treatment within different solutions.

The acidic function of the artificial saliva solution (neutral, acidic and basic units) for different periods of time (0-120 hours) using SEM electron microscopy.

The EDS analyzer was very useful in understanding and assigning the initial distribution of primary or constituent substances. The results of EDS showed all the components formed on the surface of the metal due to corrosion from different pH (0-8), pH (neutral, acidic, basic units) and types of artificial

salivation (0-120 hours). An overview of the surface of the overlapping substance of amalgam/ PVA is shown in Figure-8 (A, B), indicating a significant decrease in the amounts of silver due to the dissolved metal in the solution that resulted in corrosion and oxidation reactions. In addition to the significant reduction in the concentration of oxygen, it indicates the presence of a thin layer of oxide film on the surface of the metal, which acts as a protective layer during corrosion in sever environmental conditions [11]. On the other hand, there are many impurities, such as carbon C, aluminum Al, and silicon Si that are due to the nature of the alloy of amalgam (silver microwaves), as well as the emergence of new elements, namely Na, phosphorus P, potassium K and calcium Ca, in various solutions of artificial saliva.

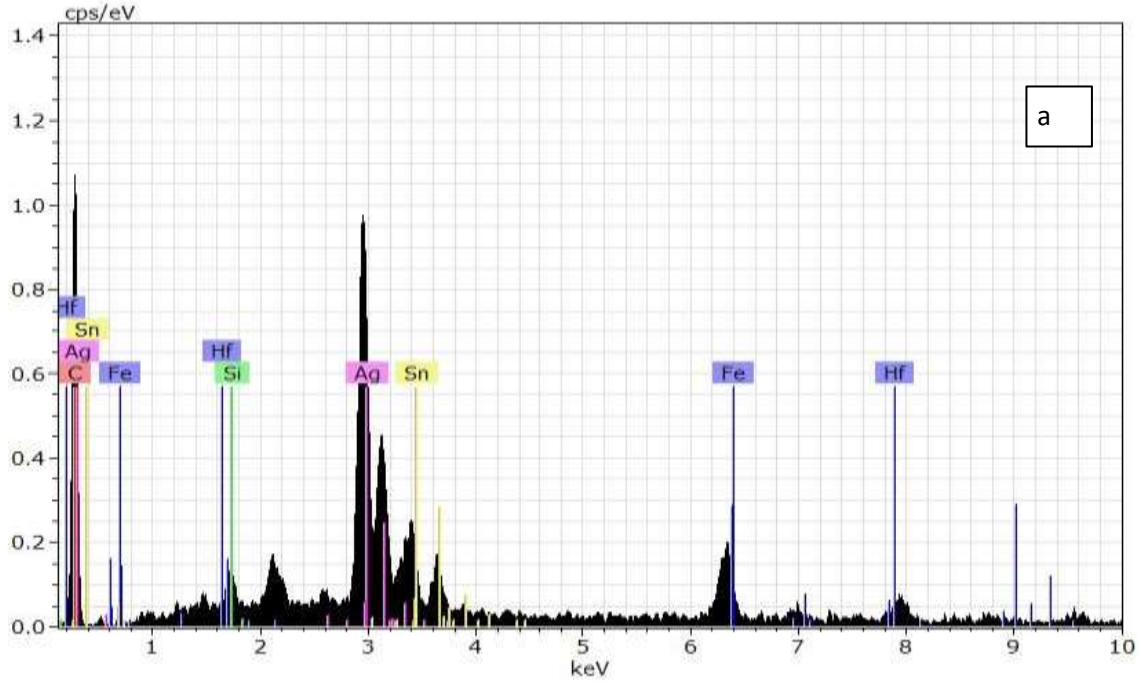

Figure 8A -X-ray diffraction measurement for optimum composite material 67/33 Mg silver/ PVA gel thermally treated before chemical treatment in different acid solutions that functioned as artificial saliva solutions.

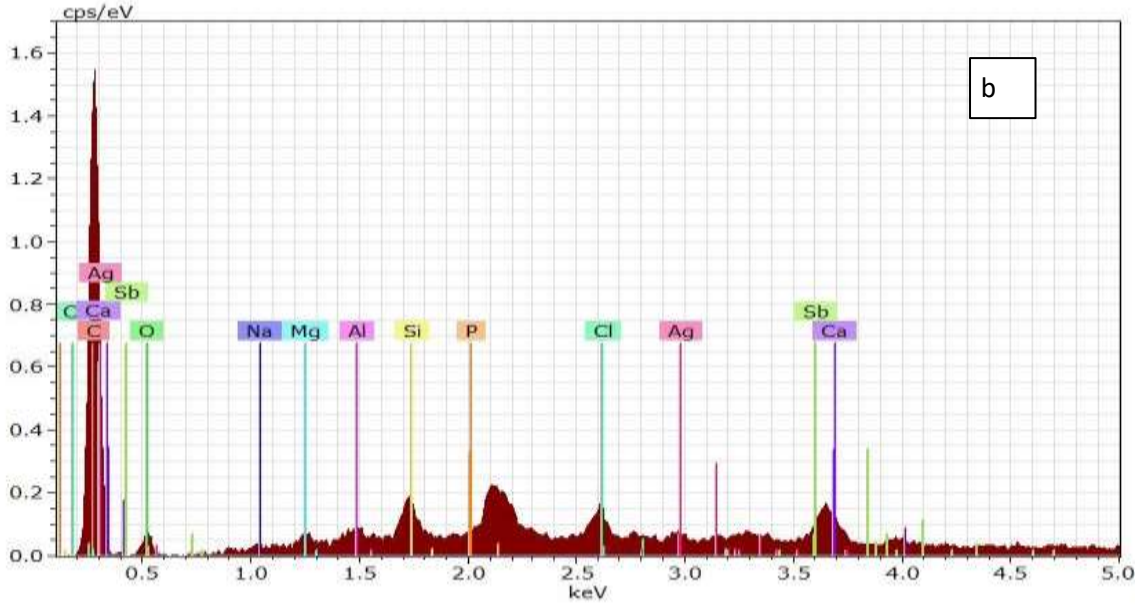

Figure 8B-X-ray diffraction measurement for optimum composite material 67/33 Mg silver / PVA gel thermally treated after chemical treatment in different acid solutions that functioned as artificial saliva solution

Conclusions

Creation of thin film oxide molecules due to corrosion reactions on the surface was detected using SEM. The diffraction of X-ray demonstrated the disappearance of certain substances, as well as the formation of the thin oxide on the surface. This layer is important for the protection of teething structures. The results showed that the pure silver fillings and the modified mixture with a medium concentration of PVA (4.8 g) and 33/67 (MAg)/ PVA was the ideal mix; this mixture was superior in the resistance to chemical changes caused by acidic solutions, which were different for artificial saliva solution, in addition to its excellent thermal and mechanical properties. These properties confer excellent chemical resistance to the various environmental conditions within the mouth, which is important for maintaining the longevity of teeth, as well as adding durability to the orthodontic strands.

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