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Consolidation of Fragile Archaeological Bone Artifacts: A review

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Abstract

Archaeological bone artifacts suffer from fragility and weakness, which are due to the effect of various factors either in the burial environment (such as moisture content, temperature, pH value, pressure, microorganisms, insects, and salts) or after extraction and exposure to surrounding environmental condition in museums, storage and excavation areas to different factors such as air pollutants, light, high temperature, relative humidity and etc. The study focuses on the most critical factors that affected bone artifacts before and after extraction from the excavation areas. It also aims to survey the common polymers used in the consolidation of bone artifacts to produce conservators in Egypt a list of polymers used with mention their advantages and disadvantages in order to choose the best ones. Various polymeric materials (natural and synthetic) were used in the field of archaeological bones in order to enhance the bone's mechanical properties and morphological structure. Archaeology scientists utilize polymeric materials in bone conservation for giving structural support to overcome the fragility and weakness of archaeological bones. Analysis and investigation become vital in the conservation field. They are used to identify the best materials used as bone consolidants, determine the types of materials used successfully in the past for treatment, and point out differences in the approaches of conservation and archaeology towards the preservation of archaeological materials. Polymer application for bone treatment should be carefully considered, according to understanding the physical and chemical interactions between the bone surface and polymer matrix. Some synthetic polymers are used by conservators for the consolidation of archaeological bone artifacts since they gave good results compared to natural consolidants, which gave many disadvantages.

Keywords: Archaeological bones, deterioration, consolidation, natural resin, synthetic polymers, analytical techniques.

1. Introduction

Archaeological human and animal bone artifacts are of great value, in order to benefit from them in many different studies. Human remains have high research value and can make a major a serious to such subjects as human evolution and adaptation, and genetic relationships, Population relationships through genetics and morphology, demography and health in the past, Diet, growth , and activity patterns, causes of death and diseases, history of disease and medicine, burial practices, beliefs and attitudes, and the variety of cultural practices in which the body and its parts are used [1]. Human remains can also represent the history and process of human evolution [2].

The excavation of animal remains is as important as the other archaeological evidence as they provide a unique insight into the behaviour of past human populations. It is the responsibility of archaeologists to provide the most accurate information possible and it is, therefore, important to consider the methods of retrieving faunal assemblages even before the excavation commences. Animals Also formed an important part of people's lives in the past and the bones from archaeological sites may provide information on not only diet but also on care, hygiene, climate, status, season of occupation, hunting methods, butchery methods, industries, trade and even religion [3].

Bone is a complex, composite material with a mineral matrix commonly thought of as carbonated calcium hydroxyapatite (CHA) which has extensive hetero ionic substitutions [4]. It originally consists of

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77% microcrystalline hydroxyapatite; the remaining 33% is primarily made up of the protein collagen (Fig. 1), non-collagenous protein, liquids, and mucopolysaccharides, as well as other components. The mineral and protein components of bone can be expected to react quite differently depending on the burial environment [5]. Type I collagen constitutes approximately 20% of bone by mass and 35% by volume, and >90% of the organic matrix of bone. There are other types of collagen. The main difference between Type I collagen in different tissues (e.g. skin and bone) relates to the arrangement and cross-linking of the collagen fibers, rather than the chemical composition of the fibers themselves [6].

However, bone mineral is rarely stoichiometric, containing many substitutions such as magnesium, sodium, potassium, fluorine, chlorine, and carbonate ions. The apatitic mineral in bone is closely associated with the collagen fibers and is made up of long, flat, plate-like nanocrystals that are approximately 40 nm long, 10 nm wide and 1-3 nm thick. This mineral component gives rise to the compressive strength of bone [7]. Macroscopically, bone is composed of two types of skeletal tissue: cancellous and compact bone. These two types of bone are distinguished primarily by the size of pores present within their structure; cancellous bone has large pores, while compact bone has few, if any, pores. Compact bone is much denser than cancellous bone. The density of archaeological bone has ken shown to be directly related to its tensile strength, and therefore its ability to survive attritional processes. These attritional processes can be either chemical or mechanical; denser bone will survive in higher frequencies [8].

Estimating the deterioration of archaeological materials is an important process in the field of archaeological conservation. It gives a good idea of the degree of deterioration, and accordingly, a successful plan can be made for the various treatments and conservation processes [10-46].

Many factors of deterioration, either in the burial environment, or the exposure environment after extraction from excavations, play a major role in the deterioration process of archaeological bones [15-17], [47-50]. All the deterioration factors of archaeological bones will be explained in detail below.

From the middle of the second half of the last century until now, many materials have been used in the consolidation of fragile archaeological bones. Natural resins such as beeswax, shellac and etc. have been used, and synthetic polymers have also been used. The consolidation materials derived from different sources will be explained in detail below. The advantages and disadvantages will be also mentioned.

This study aims to:

- Explain the factors and mechanisms of archaeological bone deterioration from different environments (burial environment, exposure environment after extraction from excavation areas).
- Conducting a survey of the consolidation materials from different sources, mentioning the advantages and disadvantages of these materials.
- mention the most important analysis and investigations used to assess the deterioration of archaeological bone artifacts, and also to evaluate the consolidation of materials used in the treatment of archaeological bones.

2. Deterioration of archaeological bones

There are some factors in the burial environment that are considered the most dangerous to archaeological bones. The speed of degradation of archaeological bones relies on types of burial environment, water content, pH, oxygen amount and etc. [50]. Moisture content of the soil, temperature, and soil composition are the most dangerous factors that affect archaeological bones. There are other factors both in burial environment, and after exposure to surrounding environmental conditions in excavation areas, storage, and museums. These factors can be summarized as follows:

2.1.1. Water and deterioration of archaeological bones

Water in and around the archaeological materials is one of the most important factors for archaeological preservation and/or change of mineralized tissues. Three major hydrological scenarios were proposed: Diffusive, recharge, and flow regimes [51]. The rate and volume of water flow through a bone in soil depends on the relative hydraulic conductivities of both soil and bone (i.e. their relative porosities), and the total volume of water available to flow (i.e. the amount of rainfall). This flow regime considers the worst situation for the survival of archaeological bones and in extreme cases can lead to total leaching of the skeleton, leaving only a soil silhouette or ‗sand body' [52], As water leaches away the organic substance of bone, and make it very fragile [53]. So bones buried in well-drained soils are particularly susceptible to leaching of the mineral.

Water also makes the bounds between organic and inorganic constituents susceptible to deterioration. When the bands weaken, more water is absorbed and breaks the bonds [47].

The remains can undergo directional deformation in each of their three axes as they respond to the relative humidity of the environment. Fractures, cracks, fissures, and streaks occur along the axes of the bone tissue as a result of the volume changes and stresses induced by these new circumstances, increasing the piece's instability [54].

Archaeological bones became much better preserved by preventing long contact with water. Deep bone fractures can be caused by repeated freezing and thawing cycles [53].

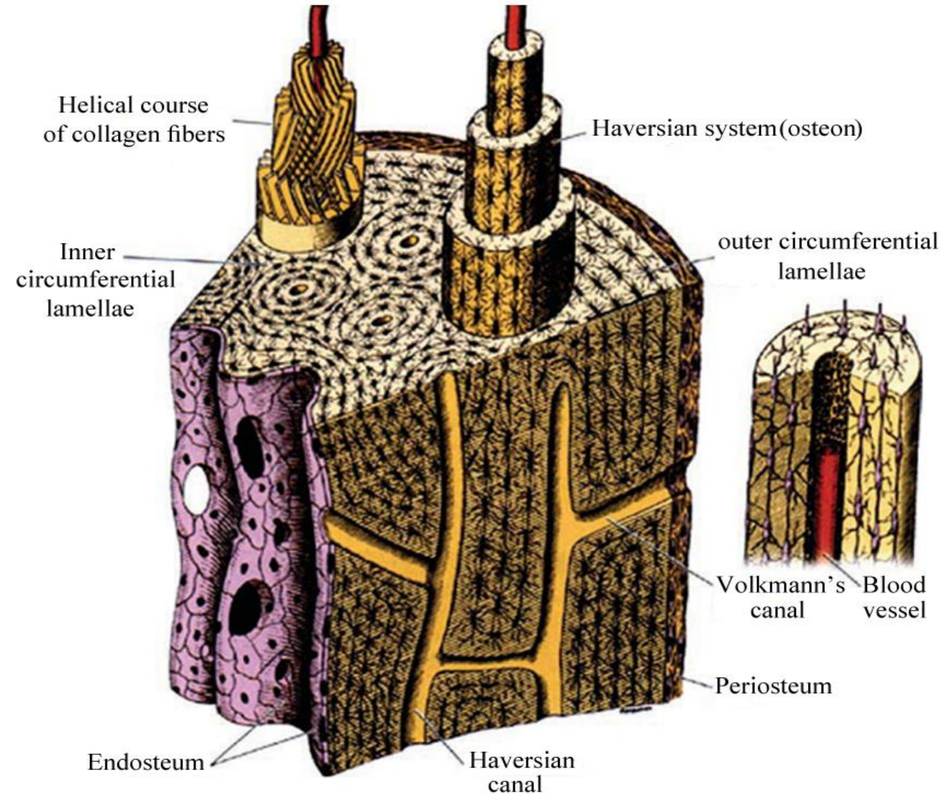


Fig. 1. Schematic drawing of the cortical bone [9].

2.1.2. Temperature

When bones are exposed to a heat source of around 300°C, they quickly carbonized, when temperatures exceed 450°C, the remaining organic carbon is progressively destroyed, But at 100° C mineral phase alternated as the water is removed. Over than 300°C carbonate content decreases [48]. Some studies ensured that heating above 700 C causes the formation of new mineral phases (e.g., formation of CaO for apatites with Ca/P > 1.67), depending on the ionic substitution of the starting apatite. However, whereas 500–650 C is the accepted temperature range required to completely remove the organic phase of bone but heating to 800 ºC may be important to eliminate possible pathogenic agents and obtain protein-free samples. Regarding the mineralogy of the sample, it is generally accepted that heat treatment promotes crystallinity of bone-derived hydroxyapatite and increases crystallite size [55].

Temperature and moisture have a great effect on how diagenesis will affect the trace-element levels in bone. The degradation of the organic component of bone and, consequently, the modification of trace elements, are both accelerated by high temperatures and high moisture levels. Depending on the type of bone, diagenesis is different. Some authors [56-58] discovered that ribs and vertebrae, which are mostly formed of trabecular bone, are more susceptible to post-mortem change than bones, which are primarily made of cortical bone (i.e. femora, tibiae, and humeri) [59].

2.1.3. Soil composition

Bone preservation varies not just from soil to soil but also from one place of burial to another burial. Soil include varying ratios of mineral and organic matter, air, and water. According on the size of the particles in the soil, it can be classified as clay, silt, sand, or gravel. Burial environment can affect bones preservation depending on pH value and types of the soil [60]. Alkaline conditions such as over limestone, as rainwater, organic acids, and biota have an impact in all soil types, so bones subjected to decay. This decay process takes time, the more soluble material decompose first, also porous bones decomposed more quickly than denser bones [61]. In general, acidic, sandy, or gravel soils are the worst for bone survival whether children bone and or adults [62], as sandy soil is easy penetrate by flood water and rains helping the migration of organic and mineral components [63]. Also sandy soil with pH lower than 5.6 caused leaching of phosphate leaving bone without trace [62].

Preservation of bones in gravel soil depend on acidity and permeability, many bones recovered from gravel soil have several chemical and physical degradation and also microbial attack resulting great increased porosity and loss of up to 90 % of collagen content [64]. At Overton Down in the UK, Armour-It was discovered that a chalk environment was not favourable for bone preservation as surface alteration of non-adult bones occurs there within a few years because their porous nature. So in chalky soil bones are exposed to eroding and becoming very weak [65].

So status of bone preservation can be affected by a number of different conditions that might exist inside burial soil. As in gravels soil Permeability, acidity, and if it's either waterlogged or anaerobic, Preservation would be good if the acidity was low. However, if the acidity is too high, the preservation will be affected [65].

2.1.4. pH value

The Scale of pH is between 1 to 14. The pH value below 7 is considered acidic, whereas any value over it is considered alkaline, and the value of pH is considered neutral at a pH of 7. The pH of most soils discovered in archaeology is between 3.5 and 8.5 on the scale. Acidic soil decomposed bones rapidly because it dissolves the inorganic matrix of hydroxylapatite. However, in an acidic environment, hydroxylapatite will breakdown into calcium and phosphorus soluble salts. A buried skeleton can last for centuries in good conditions if the soil pH is neutral or basic. It is clear that in a corrosive soil environment, irrespective of taphonomy, the effect will be the same: mineral dissolution [57].

2.1.5. Microorganism deterioration

The most common cause of bone degradation, which can happen quickly after death, is microbial destruction, besides other factors such as temperature, time, and the pH of the environment. All these factors affect the rate of collagen lost. Bacteria produce enzyme of collagenase, which breaks down bone collagen and creates a pathway for the invading organism. Microbial attack causes dissolution, which focuses in distinct zones of destruction known as microscopic focal destruction (MFD). MFD is caused by organisms from burial environments which penetrating the canal wall of buried bone and attacking the osteon tissue. Microbes such as fungi and bacteria demineralize bone, causing "tunnels" or "borings" [66].

2.2. Deterioration of archaeological bones after excavation

Some other factors affect the bones after their extraction from the excavation areas. These factors are fluctuations in temperature and relative humidity, which cause some cracks and weakness in archaeological bone [5]. Exposure to excess light for long times leads to the bleaching of bone. Light is also considered a source of heat, which leads to the acceleration of the chemical reaction and change of bone color [5, 67]. Archaeological bone and ivory in museum and storages environments respond to changes in the surrounding environmental conditions by expanding and contracting, which results in cracking, desiccation and splitting. This was due to that bone and ivory are both anisotropic and hygroscopic. As a result, moisture changes exert stress on their physical structure, causing damage and deterioration (68).

Air pollution also affects archaeological bones. The acidic condition affects both organic and inorganic phases of bones. Some erosion and pitting can be obtained from the effect of the acidic conditions of archaeological bones. Microorganisms (fungi and bacteria) affect archaeological bone in exposure environment after excavation.

The weakness and fragility of archaeological bones are considered the most common aspects of deterioration. Accordingly, archaeological bones need consolidation in order to increase their strength and durability [69].

3. Consolidation of archaeological bones

Consolidation is a set of action which aimed to strengthen fragile materials [70]. It wasn't undertaken to strengthen the fossil only, but to protect it during treatment [71].

In archaeological fields a lot of consolidation materials have been used to preserve the morphological structure of bones. Archaeologists and conservators have devised techniques and materials for using these polymers to give structural support to deteriorating and fragile bones [72]. It can be added that the application of polymers improved the properties of bones and increase their durability [31]. The mechanical and aesthetic properties are also improved [73]. consolidation give fragile, deteriorated material more structural strength [72].

It should be mentioned that there are some requirements that should be found in the polymers used in the consolidation process of archaeological bones. The most requirements are that they should have reversibility. They should be colourless, transparent, have a pH value similar or close to the pH value of archaeological bone, gave an improvement with low concentration. They should be non toxic and safety for the conservator and the object [74-75].

3.1. Natural materials

3.1.1. Natural waxes

3.1.1.1. Bees wax

Bees wax is a natural polymer and biological material made up of a variety of components (fatty acid esters, alcohols, acids, etc.). It is non-toxic and low cost. Furthermore, it is a chemistry-stable and water-repellent material [76]. It is obtained from Apis mellifica and consist of Myrical palmiate ($C_{15}H_{31}COOC_{30}H_{61}$), hydrocarbons, fatty acids and Alcohol The melting point of Bees wax is at 62 ˚C -64 ˚C. Beeswax was commonly used in the earlier of the twentieth century as a lot of natural consolidation materials [77]. It was used in consolidation of archaeological bones in 1924, but it has a lot of disadvantages such as poor stability, low penetration through the fibre structure and can cover the surface only, which can effect on the investigation of the surface morphology of the bones [78].

3.1.1.2. Paraffin wax (petroleum Wax)

Paraffin wax is a macrocrystalline wax composed of long-chain hydrocarbons and is obtained by petroleum distillation [79], with the chemical formula Cn H2n+2 with $n \geq 16$ [80]. It was widely used in archaeological sites as a lifting material in the nineteenth and twentieth centuries by pouring molten liquid over objects [79]. Paraffin wax is odorless white translucent solid and tasteless. It also affects the dating of the archaeological object giving a date much too old [72]. The density of paraffin wax is 0.88-0.941,2 (g/cm^3), and its Melting point is 47-65 °C [79].

3.1.2. Natural resin

Natural resin is classified according to its sources. There are two sources of natural resin. It may be derived from animal sources or from plants. Rac or shellac is the only one animal resin, but there are a lot of resins from vegetables or (plant-based resin) such as dammars and balsams. There are others derived from mineralized or fossilized as amber and copals [82].

3.1.2.1. Dammar resin (Triterpenoid resin)

Dammar resin consists of dammarolic acid C45 $H_{77}O_3(COOH)_2$ [83]. It is a natural plant resin. It refers to a group of resins produced by trees of the Dipterocarpaceae family, which are mostly found in eastern India, Indonesia, and Thailand. Dammar is readily scratched and has little flexibility in its unprocessed solid form. Despite these disadvantages, it was widely used due to its ability to dissolve in a wider range of solvents [84].

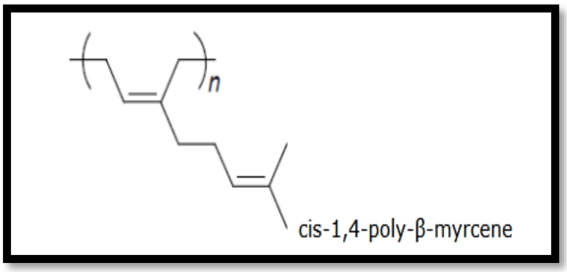
3.1.2.2. Colophony (Rosin)

Rosin, is also known as colophony. It is a non-volatile conifer tree resin component, which is solid and brittle. Gum rosin and tall oil rosin are two industrially important forms of rosin that may be identified based on their origin [85].

Rosin is composed of complicated composition that contains monoterpenes, (such as pinene, limonene, long- folate, and caryophyllene) sesquiterpenes, and diterpenes (such as resin acids), in which resin acids (RAS, ($C_{19}H_{29}COOH$) being the most important component [86]. It is typically unsuitable for use in current adhesive systems because it is subject to oxidation and other reactions, as well as crystallization. [87].

3.1.2.3. Pistacia lentiscus (Mastic)

The polymer of mastic was recognized as cis-1,4-poly-β-myrcene [88]. Mastic gum is obtained from the Pistacia lentiscus tree, which is only found on the Greek island of Hios [89]. This resin appears to be made up of a number of organic components, including a natural polymer, volatile and aromatic components that make up the essential oil (Mastic oil), terpenic acids, phytosterols, polyphenolic molecules, and a lot of other potentially active secondary metabolites, some of which have been isolated and determined for the first time in nature [90]. The formed film of mastic became brittle in a short time. It is par partially dissolved in all organic solvents and completely dissolved in polar solvents [84].



Polymer of mastic [90]

3.1.2.4. Shellac (hydroxy-6-(hydroxymethyl)-6-methyltricyclo [5.3.1.01,5] undec-8-ene-2,8-dicarboxylic acid;9,10,15-trihydroxypentadecanoic acid)

Shellac is a thermoplastic resin produced from the secretion of the lac bug (Kerria lacca), which is mostly found in India. This biopolymer is made up of natural singles, aliphatic acid polyesters, and sesquiterpenoic acid polyesters [91].

It consists of both polar and non-polar components. And it is a low-cost, non-toxic alternative. However, as a natural material, it is susceptible to many factors that might lead it to become brittle over time [38]. It was commonly used in dense layers, producing surface gloss and affecting coloration (Fig. 2). Over time this thick layer became unstable and forms a hard layer that cracks and begins to peel and fall off of the surface of bones [54]. It was for the consolidation of archaeological bone (Fig. 2).

Fig. 2. Example of consolidation of bone material with Shellac. Left: left lateral view; Right: ventral view. Note the surface gloss and differences in color [54].

3.1.2.5. Animal glue (Gelatin)

Animal glues are natural polymers obtained from fish collagen or mammalian collagen which is the main structural protein component of skins, cartilage, connective tissue, and bones [92]. Collagen is made up of long protein molecules composed of naturally occurring amino acids that are connected in a precise sequence by strong covalent bonds. It is insoluble in cold water. It convert into gelatin in different temperature condition according to its source (mammalian or fish glue). However, when collagen is heated to extremely high temperatures (such as 80-90ºC), it loses its gel strength [93].

animal glue was used as consolidant for archaeological materials, but it has some disadvantages. The protein cross link in animal glue can promote by temperature, humidity, and light, which affected peptide bond oxidation [91]. It can be added that animal glue is susceptible for the growth of microorganisms with improper conditions.

When choosing glue, conservators should be aware of the effects of pH in a substrate, as this chemical property varies depending on the glue type and treatment used during manufacture. Mammalian glues tend to be more acidic, but fish glues are more neutral, while pure gelatines from mammals and fish have pH ranges of 5.0-6.5 and 3.5-5.0 respectively. Other mechanical and physical qualities are affected by this factor [93].

Traditional glue was used in a human skeleton that was recovered during field excavation in the year of 1982 in a pit at the archaeological site of Al-Kharaneh, Al Azraq desert, 70 Km east of Amman (Fig 3). It appears to use gum to glue vertebrae together without cleaning and the application method was not thin films (Fig. 4) [95].

Fig. 3. Previous conservation process in which the glue adhesive material was used excessively, it was used to adhere vertebra broken parts together [95].

Fig. 4. The use of gum to glued bones together without cleaning [95]

Most of the resin materials mentioned above are affected by the improper conditions in museums or storages (such as excessive exposure to light, fluctuation in relative humidity and temperature, contaminations, etc.). These conditions lead to yellowing and cracking of these resins. It was concluded that both moisture and other contaminants may lead to a reduction in joint strength and catastrophic adhesive failure [96-97].

3.2. Synthetic materials

Synthetic materials include Synthetic waxes and Synthetic resin.

3.2.1. Synthetic waxes

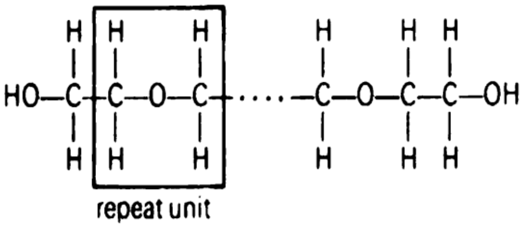
3.2.1.1. Microcrystalline wax

Microcrystalline waxes are predominantly constituted of branched saturated hydrocarbon chains, Chemically it's composed of complex mixtures of paraffinic, isoparaffinic, and naphthenic hydrocarbons.

It melts at temperatures ranging from 60˚ to 94˚ and is derived from petroleum distillation fractions [98]. There are a lot of types of microcrystalline waxes as Be Square™ 195, Cosmolloid® 80H, and Multiwax® W-445.

3.2.1.2 . Polyethylene Glycol Wax (PEG)

It is a long-chain polymer with a structural formula $HOCH_2(CH_2OCH_2)_n\ CH_2OH$ [99]. Polyethylene glycol (PEG) is known as poly (oxy ethylene).



Chemical composition of polyethylene glycol [99]

Polyethylene glycol (carbowax) is a water-soluble wax, as it can be melted or dissolved in warm water. Also, it is a colorless, nontoxic, and flexible material [100]. One of its disadvantages is that the removal procedure requires picking, melting, wiping with ethanol, or dissolving in hot water to assure complete removal which can affect the object's surface [101].

Carbowax is used as a consolidant nowadays, but it degrades over time and cannot preserve the findings for a long duration [102]. It may be necessary to make temporary support embed fossil from carbowax, if it is very fragile, as conservator makes abed of wax that hold the fossil in to stabilize it. Carbowax gives good support to weak and delicate bones [100]. It was reported that polyethylene glycol has often been used as the consolidant of choice because it is relatively reversible, stable, and inexpensive [103].

3.2.2. Synthetic resins

Synthetic resins are divided into three main parts according to their use in the conservation and restoration field: thermoplastics resin, thermosetting resin, and coldsetting resin.

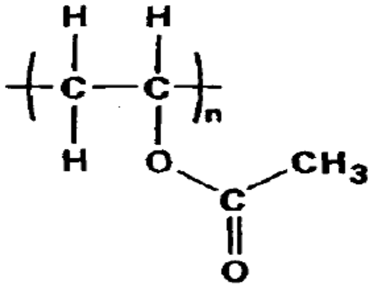
Thermoplastics are two-dimensional molecules that can be melted by heat. Thermosets are three-dimensional networks of materials that cannot be altered by heat [104]. It includes urethanes, phenolics, amides, polyesters, epoxies, and amino resins. Because of their extreme durability over periods of time and their flexibility to be adapted to industrial coating procedures, they have achieved extensive popularity in the industry [82].

Coldsetting resins are usually used in two components, which are resin and hardener [105].

3.2.2.1. Polyvinyl acetate (White glue)

One of the most extensively used adhesives, poly(vinyl acetate), It is found in conservation applications such as consolidation and coating. It is a chemically stable thermoplastic that may be easily reversed by dissolving joints. PVA is brittle, like cellulose nitrate, but a little quantity of solvent is kept for a long period [106].

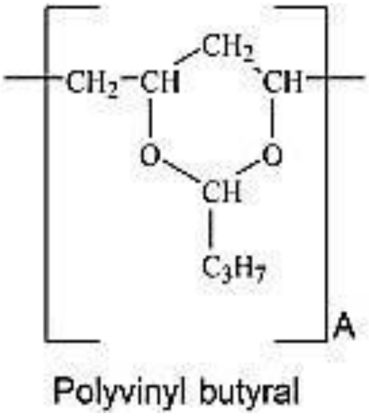
However the common use of polyvinyl acetate, practically there are three main factors that work against using this resin. Firstly changes in solubility due to aging, secondly the impossibility of removing this polymer from the pores of deteriorated materials, and finally, the risks which find in removing the resin from original materials [70].



Chemical structure of poly (vinyl acetat) [107]

3.2.2.2. Polyvinyl Butvars (PVB) (Mowital)

Polyvinyl butyral is a random terpolymer that contains butyral, hydroxyl side groups, and a small number of acetate units [108]. It belongs to the family of poly acetal vinyl resins that are formed from the reaction between aldehydes and alcohols. PVB is a thermoplastic resin. It is colorless, amorphous [109], and dissolved in Alcohol, glycol ethers, and a mixture of polar and nonpolar solvents [110]. It is used commonly in archaeology and paleontology because it's resistant to aging [33]. The use of polyvinyl butyral in consolidation has some disadvantages such as insufficient penetration and unsatisfying mechanical strength [111].



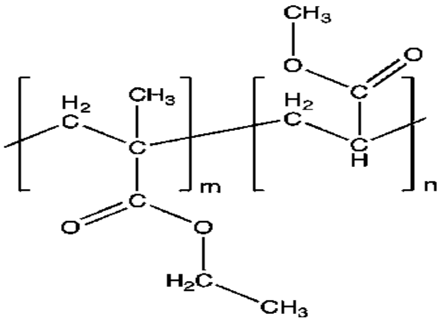
Chemical composition of Polyvinyl butyral [112]

3.2.2.3. Acrylic Resins

There are many types of acrylic resin on market such as Paraloid® B44, B48N, B66, B72, and B82 [113]. Acrylate resins are common with Low solvent evaporation rates [114]. Because of the mechanical qualities and the simplicity of application of paraloid, it is one of the most commonly acrylic resins in the consolidation of archaeological artifacts.

3.2.2.3.1. Acryloid B72 (Poly (methylmethacrylate: ethyl acrylate) 50:50)

Acryloid B72 (paraloid P72 in Britain) is a copolymer of methyl methacrylate and ethyl acrylate [72]. It has a lot of advantages such as good adhesion strength, light resistance, transparency, mechanical resistance, and reversibility [115]. It dissolves in acetone, ethanol, and another solvent [54].



Chemical structure of Paraloid B72 [116]

Dissolving paraloid in acetone avoids using water allow to keep impregnation under control because of acetone volatility [117]. However Paraloid P72 has a high cost compared with other commercial adhesives [118], it considers one of the best adhesive materials in the conservation of bones as it is used in gluing broken parts of archaeological bones because it can be used under room temperature [95].

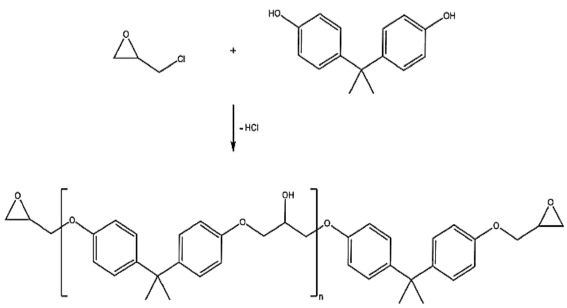
Paraloid B72 has some disadvantages, such as its incompatibility with wet substrates and a humid environment. It has a glass transition temperature of 40 ◦C, which could be an issue when consolidation treatments need to be applied in situ. In an outdoor environment, the change of color, and alteration of the transport properties of the substrate can occur. It is sensitive to UV radiation, and it has poor outdoor durability [119].

3.2.2.3.2. Paraloid® B44 (poly-methyl methacrylate: ethyl acrylate: butyl methacrylate) 70:28:1

Paraloid® B44 is also known as (poly-methyl methacrylate) (PMMA). Many studies have examined the Paraloid® B44 copolymer resin, which is made of poly ethyl acrylate (PEA), polymethyl methacrylate (PMMA), and ethyl acrylate (EA). These studies revealed that the crystals are transparent and not yellowish and have good resistance to UV radiation, corrosion, oxidation, and resistance to external factors [120].

3.2.3.4. Polyepoxide (Epoxy Resin)

Epoxy resin, like other epoxies, is a copolymer composed of an epoxy "resin" and a polyamine "hardener." The resin is made up of monomers or short-chain polymers with an epoxy group at the end [121]. It is widely used in industry but has rarely been used in conservation because it tends to turn yellow by exposure to heat or light and it is not easy to remove [122].



Formation and structure of an epoxy resin prepolymer [121]

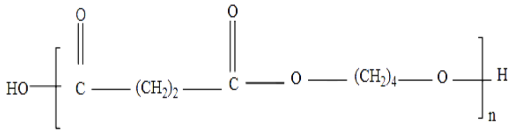
3.2.3.5. Poly (hexyleneadipate) (PHA)

PHA was synthesized by combining equal moles of 1,6-hexanediol and adipic acid with 0.4 mol percent p-toluene sulfonic acid. Dimethylbenzene was used as a solvent. This reaction was carried out in a nitrogen atmosphere with continuous agitation at the refluxing temperature of dimethyl benzene (143°C) [123].

3.2.3.6. Poly (butyleneadipate / succinate) or (PBS)

PBS chemical formula is $HOOC(CH_2)n\ COOH$ n=3. Its copolymers are a family of biodegradable polyesters produced from succinic acid, butanediol, or other dicarboxylates and alkyl diols [116]. PBS is a white crystalline thermoplastic with a density of 1.25 g/cm 3 and a melting point (Tm) ranging from 90°C to 120°C, as well as a low glass transition temperature (Tg) ranging from 45°C to 10 °C. It has excellent mechanical properties and processability in general. It's like other aliphatic polyesters, and is thermally stable up to roughly 200 0C.

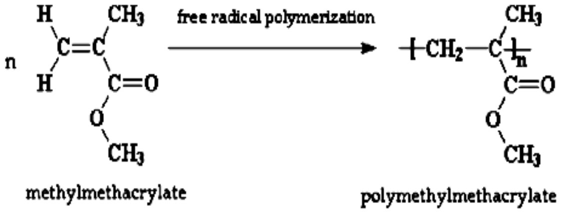
PBS is now synthesized using the condensation polymerization of petrochemical succinic acid and butanediol, both of which are typically obtained from maleic anhydride [124].



Poly butylene / succinate repeating unit [124]

3.2.3.7. Polymethylmethacrylate

Polymethylmethacrylate (PMMA) ($C_5O_2\ H_8$) is an acrylic resin produced from the monomer methylmethacrylate (MMA) and produced by a variety of polymerization mechanisms. Among many different resins, PMMA is widely used because of its high colorability, attractive appearance, good weather resistance and mechanical strength, acceptable chemical resistance, and great transmission of around 93%. The interconnected porosity, bone on, and in-growth characteristics are the main benefit, allowing it to be used as bone cement to remodel missing bone [125].

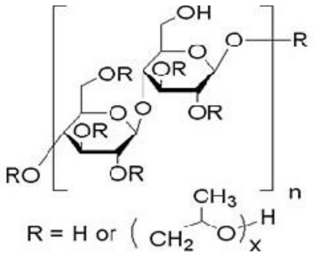


Preparation of the polymer PMMA [126].

3.2.3.8. Polymer derived from cellulose

3.2.2.8.1. Hydroxypropylcellulose (HPC) **(Klucel)**

Hydroxypropylcellulose (HPC) is sold under the trade name “Klucel”. It is non-ionic cellulose and it can be solved in water (below 40˚C) and organic solvent (ethanol) [127].

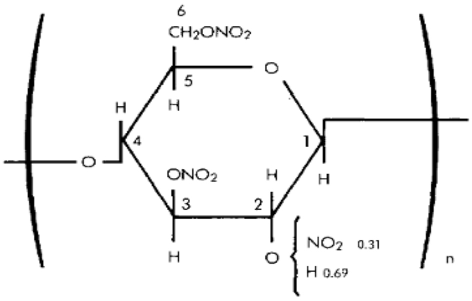


The structure of hydroxypropyl cellulose (HPC) [128]

The type of Klucel G was the most common of Hydroxypropylcellulose for the treatment of organic materials such as bone artefacts. The application of low concentration from this polymer is better than high concentration, because it allow it to penetrate through the fibre structure of bone.

3.2.2.8.2. Cellulose Nitrate Resins

The polynitrate ester of the natural polysaccharide, cellulose, and for a polymer averaging 2.3 nitrate groups for each glucose unit has the structure is called cellulose nitrate [129].



Cellulose nitrate structure for polymer with a 2.31 degree of substitution [129].

Cellulose nitrate was first marketed as Celluloid in the United States, it was cheap to manufacture, also resilient, and water - and acid- resistant, but it has a lot of disadvantages as it becomes yellow and brittle, shrinking, warping, crizzling, and releasing hazardous gases over time at room temperature [130].

Previous treatments had a negative impact on artifacts, causing them to be completely destroyed. Artifacts that perished in the past quickly after their construction because the material had entered into the voids or cells of organic materials and caused total disintegration owing to chemical interactions between the artifact’s composition and the treatment materials [131].

In archaeology, the fragility of cellulose nitrate adhesives was recognized as early as 1936. Celluloid substances "become brittle and flake away. Many museum examples show damage caused by adhesive failure when the adhesive has pushed the ceramic paste away where it was applied (Fig. 5) [72].

Fig. 5. Shrinkage of aged polymers can pull apart the structure of bone. The bone surface in the center of the photo has been pulled away by the dark adhesive (probably shellac) [72]

4. Analysis and investigation used to evaluate bones consolidation materials

In the conservation field, analysis and investigation are very important. They evaluate the efficacy of conservation materials and techniques and reveal their advantages and disadvantages [33]. FTIR spectroscopy is a semi-quantitative method for identifying the compositional and structural properties of materials by characterizing bond vibrations absorbed at specific wavelengths of transmitted incident light from infrared radiation. It is very important in the evaluation of conservation material When used on bone, FTIR spectroscopy may reveal the existence and quality of preserved organic components, as well as the size, structural order, and strain of bioapatite crystals. The infrared splitting factor (IRSF) can be used to determine the crystallinity of a sample, it extrapolates the changing size and order of bioapatite crystals through increased splitting visible in the PO43- v4 peaks. FTIR spectroscopy has also been successful in detecting thermally changed bone such as bone composition. Several heat-induced peak transformations can be used to detect bioapatite crystallinity [132].

The crystallographic structural properties of the mineral component of bone can be determined using XRD (X-ray diffraction) [55].

X-ray fluorescence is a method for determining the presence of exogenous materials in bone samples in order to evaluate the skeleton's preservation status and indicate the chemical damage to bones [95].

A microscope was used to investigate the surface morphology of bone, which revealed more information about the status of the surface (Abdel-Maksoud and El-Sayed, 2016). Scanning electron microscope (SEM) micrographs depicted the change in morphology of bone, Also EDAX study identifies the Vital elements in bone, such as calcium (Ca), phosphorous (P), carbon (C), and oxygen (O) [133]. A light microscope was used to detect current evidence of specific heat alterations and changes in bone histology [48].

Another analytical method for detecting crystallinity changes is Raman spectroscopy. The sample is irradiated by monochromatic radiation via laser sources that can operate in the UV, visible, or near-infrared spectral region (785 to 1064 nm) [134].

We should be aware that some consolidant materials can affect the analysis of bones. Stable isotope analysis, trace element analysis, scanning electron microscope surface investigation, DNA recovery, and determination of specific gravity are just a few of the analytical procedures that might be affected by consolidation. Furthermore, studies show that acrylics and poly(vinyl) acetate polymers may invalidate various biochemical laboratory tests. The addition of carbon to the sample in the conservation process would alter any date obtained from radiocarbon. Paraffin wax (petroleum-based material) gives a too old date , but synthetic and natural resin give the reverse [72].

Carbon dating, often known as radiocarbon dating, is a technique for estimating the age of organic material by utilizing the characteristics of the radioactive carbon isotope 14C. Any date derived using radiocarbon dating would be false if carbon was added to a sample through preservation. Paraffin wax and other petroleum-based compounds would give a date much too old, whilst natural and synthetic resins would give the opposite result. So Bone that may be used for radiocarbon dating shouldn't be treated with consolidants [72].

Analysis of the carbon and nitrogen stable isotope ratios has several applications in ecology, plant physiology, and geochemistry. Archaeology uses all this information to learn more about earlier human subsistence practices and environmental conditions. The δ13C and δ15N values of bone collagen provide a direct and quantitative measurement of protein food intake in both animals and humans.13C values can distinguish between the consumption of terrestrial and aquatic resources [135].

5. Conclusion

Archaeological bone artefacts in excavation areas, museums and storages suffer from deterioration, which was due to some factors in the burial environments such as moisture content, temperature, pH value, microorganisms, insects, etc., or some other factors found in surrounding environmental conditions in museums and storage such as light, fluctuation in relative humidity and temperature, air pollutions, bad ventilation, and etc. The most common aspects of deterioration of archaeological bone artifacts in different locations are weakness and fragility. The consolidation process of archaeological bones became vital in the conservation field. Many polymers which have been derived from natural or synthetic sources have been used for the consolidation of archaeological bones. Examples of natural materials are natural waxes such as bees wax and paraffin wax. Natural resins such as dammar resin, rosin, mastic, shellac and animal glue have also been used. Synthetic materials were also used for the consolidation of archaeological bones. Examples of these materials are synthetic waxes such as microcrystalline wax and polyethylene glycol wax. Synthetic resins have also been used for the same purpose. Examples of these resins are Polyvinyl acetate, Polyvinyl Butvars (PVB), Acryloid B72, Paraloid® B44, Epoxy Resin, Polymethylmethacrylate, Polymer derived from cellulose such as Cellulose Nitrate and klucel. The synthetic polymers had good advantages compared to natural polymers. This study confirmed that conservators should know the properties of the polymers used and the possible chemical and physical interaction between the polymers and archaeological bones. This study also confirmed that the analysis and investigation are very necessary for the determination of the state of conservation of archaeological bones in different locations. They are also necessary for the evaluation of the efficiency of polymers for the consolidation of archaeological bones. There many types of analysis and investigation that can be used for archaeological bones such as FTIR, XRD-EDAX, SEM, and etc.

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