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Sustainable future alternatives to petroleum-based polymeric conservation materials

**YVONNE SHASHOUA\*** 

National Museum of Denmark Kongens Lyngby, Denmark yvonne.shashoua@natmus.dk KATJA JANKOVA ATANASOVA Technical University of Denmark

Kongens Lyngby, Denmark kjaa@dtu.dk

# **CLAIRE CURRAN**

ICA Art Conservation Cleveland OH, USA ccurran@ica-artconservation.org \*Author for correspondence

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### ABSTRACT

The research described here is the first study on the use of sustainable, plant-based biopolymers in conservation practice. Two applications of biopolymers to conservation were investigated - in commercial bioplastics as substitutes for petroleum-based plastic packaging, and in novel adhesive and coating formulations. Bio-polyethylenes, bio-polyesters and bio-cellulose-based products were evaluated against petroleum-based materials. Bio- and petroleum-based polyethylenes shared optical, chemical and thermal properties. Bamboo and sugarcane fibre containers were also chemically stable. Polyester polylactic acid (PLA) bags and containers became brittle and opaque at a relative humidity (RH) above 65%. FTIR spectroscopy and thermogravimetric analysis suggested that PLA hydrolysed to produce acids. PLA/cornstarch bags fragmented on ageing and formed a gel at high RH levels. A 5 wt% solution of adhesive prepared from soya protein was an effective and reversible adhesive for wood, paper and glass, but adhered poorly to polyethylene and poly(methyl methacrylate). Humic acid-based solutions formed cohesive films which adhered well to glass, paper and soil.

# **INTRODUCTION**

Conservation treatments often employ petroleum-based plastic materials as packaging, adhesives and coatings that are synthesised from nonrenewable crude oil, a resource at risk of exhaustion within the next 100 years. The Going Green conference held at the British Museum in April 2009 concluded that conservators are under increasing pressure to review their practices in light of international environmental targets and the rising costs of fossil fuels. Biopolymers are considered sustainable either because they are synthesised from renewable sources or because they biodegrade to CO<sub>2</sub> and H<sub>2</sub>O in soil and water after use. The range and quality of bioplastics have increased dramatically since 2006 and, today, polyethylenes, polyesters, polyurethanes and polyvinyl alcohols can be fully synthesised from biomass, although their commercial availability is more limited in Europe than in the USA and South America. While extensive research has been conducted into the rates and mechanism of degradation of bioplastics on disposal (Rani et al. 2012), few projects have focused on their chemical and physical properties during use and none have addressed the application of bioplastics to conservation practice.

All materials used to conserve museum objects must be selected with a view to complying with the International Council of Museums' (ICOM) code of ethics, which states that 'original surfaces must not be visibly, physically or chemically changed by conservation'(ICOM 2013). In practice, the risk of changing original surfaces chemically can be minimised by ensuring that conservation materials neither contain nor produce acidic or corrosive degradation products. The polymeric packaging films which both satisfy ICOM's code and conservators' requirements include polyolefins, polyesters and cellulose-based materials. Polyolefins are selected for their flexibility, water and chemical resistance and low cost. Transparent low-density polyethylene (LDPE) bags are used to store samples or to isolate objects from their surrounding environment. High-density polyethylene- (HDPE) bonded textiles protect paper, costumes and wooden objects from damaging ultraviolet radiation while allowing transport of water vapour. Polyesters are selected for their glass clear transparency and their low permeability to vapours and gases, and are therefore the materials of choice for protecting documents and photographs and constructing anoxic microclimates (Shashoua and Skals 2004, Dyer et al. 2011). Cellulose-based materials absorb moisture

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SUSTAINABLE FUTURE ALTERNATIVES TO PETROLEUM-BASED POLYMERIC CONSERVATION MATERIALS and provide physical protection, and include tissue paper with and without alkali buffers. The purpose of this research was to establish whether commercially available plastics synthesised from renewable sources could provide equivalent or superior alternatives to petroleum-based polyolefin, polyester and cellulosic materials used in conservation in terms of their optical, chemical and physical stabilities.

In addition to finished bioplastic products, biopolymers are also commercially available as raw materials for further development. Water-based adhesive formulations were developed by the authors based on hydrolysed soya protein powder, while biodegradable barrier coatings to protect against contact with ultraviolet light and oxygen, developed originally for agricultural applications, were prepared from humic acid – a complex mixture of acids derived from biodegradation of dead organic matter that behaves functionally as a dibasic acid (Hualian 2012). Because photo-oxidation is the main degradation reaction for organic heritage materials, water-based, humic acid coatings were of interest to the authors. These were evaluated for their appearance, long-term stability, reversibility and effectiveness to adhere to paper, wood, cotton cloth, synthetic textiles and plastics.

## **EXPERIMENTAL**

### Materials

All materials evaluated in this research project are shown in Figure 1. The petroleum-based packaging plastics evaluated in this research were: a) low-density polyethylene (LDPE) Minigrip Plain Premium Red Line resealable zipper bags; b) high-density polyethylene (HDPE) Tyvek UV Soft Structure Style 1460 textile; and c) polyethylene terephthalate (PET) Melinex 401 optically clear film. Selection of bioplastics for evaluation was based on availability in Europe in addition to their chemical properties,



**Figure 1.** Petroleum-based and bioplastics evaluated: (upper row, left to right) circular PLA food container, fibre box, rectangular PLA food container; (middle row, left to right) Tyvek UV Soft Structure (HDPE), zipper bag (LDPE), PLA/cornstarch bag, Melinex triangles (PET), polyvinyl alcohol film (not examined in this research); (lower row, left to right) bio HDPE, bio LDPE, dried soya adhesive, dried humic acid coating

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**Figure 2.** Fish glue (upper row) appeared more yellow and was more brittle than soya adhesive (lower row) when painted onto glass slides at the same concentrations. Soya adhesive foamed more than fish glue

and comprised colourless LDPE (EB 853/72) and HDPE (SGM 9450F) granulates containing antioxidants and other components from which to extrude carrier bags. The polyethylene polymers were synthesised from sugarcane ethanol (supplied by Braskem, Brazil). Granulates were tested as supplied. Polylactic acid (PLA), the second highest consumed bioplastic worldwide, used primarily in 3D printing, is a biodegradable thermoplastic aliphatic polyester derived from cornstarch, tapioca or sugarcane. It was evaluated in the forms of circular and rectangular transparent food containers (P700) from Enviroware, UK and, combined with cornstarch and colouring agents, as a carrier bag. A food container comprising cellulose fibres derived from sugarcane, bamboo and reeds by Enviroware was also evaluated.

A bio-adhesive was prepared by adding 5 g of potato starch and 0.3 g of defoamer (tripropylene glycol monomethyl ether) to 50 mL of distilled water and heating to 65°C with magnetic stirring. After 30 minutes, 17 g of SoBind Balance LVL, a highly hydrolysed soya polymer powder from DuPont Solae, was added gradually and stirred vigorously for an additional 20 minutes. The resulting mixture had similar colour, density and texture to pouring cream and was further defoamed using an ultrasound bath. The cooled adhesive had a pH of 6.8 and was stored in a refrigerator until required.

A bio-coating was prepared by adding 27 g of a 5% aqueous solution of bio-polyvinyl alcohol (97.5%–99.5% hydrolysed) to 50 mL of distilled water while stirring. Sufficient potassium hydroxide solution (5%) was added to increase the pH to 9. Alkali conditions were required for the humic acid to disperse evenly in the polyvinyl alcohol solution. Black, solid humic acid (0.8 g) was added gradually, followed by an equal mass of hydroxymethyl propyl cellulose. The dark brown mixture was held at 80°C for 30 minutes. Dried, free films of soya adhesive and humic acid coating with a film thickness of 1–2 mm were prepared for testing by brushing the solutions onto glass microscope slides and allowing them to dry for 7 days at ambient temperature before removing. All raw materials were supplied by Sigma-Aldrich.

## Properties of bio-adhesive based on hydrolysed soya protein

The biomaterial's adhesive properties were evaluated by preparing edgeto-edge or overlap joins on new, clean samples of: wooden popsicle sticks made from white birch; Whatman cellulose filter paper (9 cm in diameter); glass microscope slides; white, Dura-Touch premium disposable vinyl gloves; LDPE zipper bags ( $60 \times 80$  mm); expanded polystyrene coffee cups; and transparent poly(methyl methacrylate) sheets ( $20 \times 20 \times 3$  mm). The adhesive properties of 5% soya adhesive were compared with a 5% solution of High Tack Fish Glue from Conservation Resources, a proteinaceous adhesive used in furniture and plastic conservation (Figure 2). Both adhesives were applied by brush to the edges of the test substrates, which were then pressed together and the materials allowed to dry while supported on polyethylene sheeting for 96 hours at  $20 \pm 2^{\circ}$ C. Soya adhesive was highly viscous and remained in place once applied to surfaces. Joins were assessed for appearance and strength using finger pull strength.

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**Figure 3.** A broken polystyrene cup was adhered with soya and fish adhesives. Soya (left) produced a less visible and more flexible join than fish glue (right)

The results (Table 1) suggested soya adhesive to form bonds of similar tensile strength but with greater extensibility and less visibility than fish glue on paper, glass, PVC and polyethylene. Fish glue formed stronger but more brittle and darker bonds than soya on polystyrene foam and poly(methyl methacrylate) (Figure 3). In addition, bonds formed between soya adhesive and substrates were readily reversed using cold distilled water or a wetted cotton swab. Reversal of bonds formed using fish glue required water heated above 40°C. The pale colouration, high extensibility, flexibility and reversibility suggest that soya adhesive has potential as a new biomaterial for conserving paper and plastics in modern art and objects.

## Properties of bio-coating based on humic acid

Although humic acid's dark colouration limited its potential applications in object conservation, the biomaterial's ability to form a cohesive film on various substrates was evaluated by applying it from a polyethylene spray bottle (300 mL) to new, clean samples of: wooden popsicle sticks; Whatman cellulose filter paper; glass microscope slides; degraded, yellowed, friable polyurethane ether foam; and potting compost. Samples were allowed to dry for 96 hours at  $20 \pm 2^{\circ}$ C prior to visual examination.

Material	Method of bonding	Adhesive	Aesthetic value (1 = poor, 5 = excellent)	Pull test to break (1 = low tension required, 5 = high tension required)
Wooden popsicle stick	Sticks were broken in two and adhered at break edges.	Soya	Adhesive invisible 5	3
		Fish	Adhesive invisible 5	3.5
Filter paper	Paper torn and edges adhered.	Soya	Adhesive slightly visible 4	Bond stronger than substrate 5
		Fish	Adhesive visible and bled into the paper causing staining 3	Bond stronger than substrate 5
Plasticised PVC disposable gloves	Fingers of gloves adhered.	Soya	Adhesive slightly visible 4	1.5
		Fish	Adhesive slightly visible through the PVC 3	1.5
Foamed polystyrene	Cup torn to remove pieces of ca. 2 cm in diameter. Pieces reattached.	Soya	Adhesive visible 3	2
		Fish	Adhesive visible as yellow-brown colour 2	5
Polyethylene bags	Lower edge of two bags were overlapped and adhered.	Soya	Adhesive visible through polyethylene bag 2	2
		Fish	Adhesive visible as caramel colour 3	2
Glass microscope slides	Two methods of adhering were investigated: edge to edge and overlapping the two shortest edges.	Soya (edge to edge)	Adhesive visible 4	2
		Soya (overlap)	Tan colour of adhesive visible Value: 2	5
		Fish (edge to edge)	Adhesive visible 4	2
		Fish (overlap)	Caramel colour of adhesive visible 2.5	5
Poly(methyl methacrylate)	Two methods of adhering were investigated: edge to edge and overlapping the two shortest edges.	Soya (edge to edge)	Adhesive visible 3	Bond dismantled by finger contact 0.5
		Soya (overlap)	2	2
		Fish (edge to edge)	3.5	1.5
		Fish (overlap)	2.5	3

### Table 1. Evaluation of soya adhesive compared with fish glue

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**Figure 4.** Accelerated thermal ageing of petroleum-based and bioplastics was conducted at high RH and 70°C



**Figure 5.** PLA food container became opaque after thermal ageing at low RH and disintegrated at high RH

The bio-coating formed cohesive, flexible films and adhered well to popsicle sticks, filter paper and potting compost. Three coats, applied with 96 hours drying time between each, were sufficient to obscure the surfaces of substrates. Single coats resisted abrasion with a polypropylene, microfiber cloth under finger pressure. Cohesive films were not formed on glass and polyurethane foam. Humic acid films softened immediately and dissolved on contact with distilled water for approximately 45 minutes. The interaction between potting compost and humic acid was noteworthy. A single application was sufficient to fix the dry soil particles in place. Particles were released again by applying water by spray. This indicates that humic acid bio-coating could be used when loose soil needs to be temporarily held in place, for example during archaeological excavations or building projects. Its simple application form, low toxicity and high reversibility in water would allow it to be removed by rain if used outdoors.

## Long-term stability of optical, physical and chemical properties

Commercial petroleum-based and bioplastic packaging materials, bioadhesive and -coating were examined for their optical, physical and chemical properties both when new and after thermal ageing. Long-term stabilities of bioplastic polyolefins, polyesters and cellulose-based materials were compared with their petroleum-based equivalents. In addition, the bioadhesive and bio-coating were examined for their film forming, adhesive and reversibility properties.

Thermal ageing was used to accelerate those degradation reactions expected for traditional polymers after 50 years in a museum display or storage area where daylight and UV radiation are minimised. Michalski (2002) suggests that a 5°C increase in temperature doubles the rate of thermal degradation of polymers in museums, thereby halving their chemical lifetimes. Petroleum-based plastics and bioplastics were thermally aged at both low relative humidity (RH) ( $20 \pm 5\%$ ) and high RH ( $90 \pm 5\%$ ) conditions at  $70 \pm 2$ °C for 60 days (Figure 4). The two RH levels selected represented the extremes of environmental conditions present in European museums, in specialist storage conditions for wet archaeological materials or for tropical collections, and were designed to accelerate both oxidation and hydrolytic reactions.

# **Optical properties and cohesiveness**

Because the plastics evaluated were intended to enclose and be in close contact with museum objects, it was important that they retained their original transparency and cohesiveness on ageing. Visual examination supplemented with digital photography was used to identify changes induced by thermal ageing. After 60 days at 70°C in low and high RH, no changes were visible for the transparent or white polyethylene materials, that is, petroleum-based LDPE zipper bags, HDPE Tyvek textile, Braskem's LDPE and HDPE, or for the off-white, opaque cellulose food container from Enviroware. While petroleum-based polyester Melinex 401 film retained its original appearance on thermal ageing, salad containers comprising PLA developed a haze after 30 days at 70°C and high RH conditions which progressed to opacity and fragmentation after 60 days (Figure 5). Orange-coloured PLA/cornstarch carrier bags disintegrated after 60 days at high

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SUSTAINABLE FUTURE ALTERNATIVES TO PETROLEUM-BASED POLYMERIC CONSERVATION MATERIALS RH. Reduction in transparency and cohesiveness with time suggested PLA containers to be unsuitable bioplastic alternatives to petroleum-based polyesters as enclosures for museum artefacts stored above 20% RH.

Free films of soya-based adhesive darkened after 30 days at low and high RH. Because the film of bio-coating was dark brown when new, no visible changes could be detected on ageing.

## **Chemical properties**

Changes in chemical structure induced by thermal ageing for all plastics were examined non-destructively using attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy. Spectra of new and aged films were recorded using a Bruker Alpha FTIR instrument fitted with a single-bounce diamond internal reflectance element with 20 scans at a resolution of 2 cm<sup>-1</sup>. Changes in physical and chemical properties of the commercial traditional and bioplastics packaging materials with temperature were investigated using thermogravimetric analysis (TGA) between 20°C and 800°C at a heating rate of 10°C min<sup>-1</sup> in N<sub>2</sub> using a Q500 analyser from TA Instruments.

ATR-FTIR spectroscopy suggested that traditional and bio LDPE were chemically identical, with characteristic CH<sub>2</sub> vibrations and deformations at 3000–2840 cm<sup>-1</sup>, 1469 cm<sup>-1</sup> and 718 cm<sup>-1</sup> (Figure 6). Both materials developed new, low-intensity bands between 1600 and 1700 cm<sup>-1</sup> after ageing at low RH for 30 days. These changes were attributed to the development of C-O groups formed on oxidation. No degradation was detected in samples aged at high RH. Petroleum-based HDPE and its bio-equivalent were chemically identical and neither material showed measurable chemical changes after thermal ageing. After 60 days of thermal ageing, only minor changes were seen in the TGA curves of traditional LDPE and HDPE with a decrease in the main degradation (DTGA) peak of 1 and 2 degrees respectively. Bio-polyethylenes exhibited similar thermal stability to their petroleum-based equivalents with bio LDPE remaining stable up to 370°C and bio HDPE up to 410°C.



**Figure 6.** ATR-FTIR spectrum of traditional LDPE (upper spectrum) and Brasken bio LDPE suggests that they are chemically identical

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SUSTAINABLE FUTURE ALTERNATIVES TO PETROLEUM-BASED POLYMERIC CONSERVATION MATERIALS ATR-FTIR spectroscopy suggested traditional polyester and PLA food containers to be chemically unchanged on ageing at low RH. Minor changes in the thermal stability of both new and thermally aged petroleum-based Melinex 401 were detected between 200°C and 400°C with 2.5 % weight loss at 600°C. Weight loss was attributed to the loss of a low-molecular weight polymer fragment or unreacted monomer. TGA suggested PLA Enviroware food containers to degrade at 280°C when new, and 4–5 degrees lower after thermal ageing, suggesting a similar thermal stability to petroleum-based PET.

PLA food containers showed absorbance bands typical of polyesters including those at 1714 cm<sup>-1</sup> attributed to C=O groups and 1240 cm<sup>-1</sup> due to C=O and C-O stretching. However, after 30 days thermal ageing, both PLA containers and PLA/cornstarch bags showed a decrease in intensity of bands between 1200 and 1000 cm<sup>-1</sup>, attributed to the breaking of C=O and C-H bonds, and an increase in intensity and broadening of peaks around 3000 cm<sup>-1</sup> due to the formation of OH bonds. Changes were attributed to the cleavage of ester bonds during hydrolytic breakdown resulting in the formation of aldehydes, ketones, lactic acid and water (McNeill and Leiper 1985). Differential scanning calorimetry (DSC) was used to further investigate PLA's degradation pathway, particularly its increasingly hazy appearance. Samples were investigated using TA Instruments Q1000 between 20°C and 200°C at a heating rate of 10°C min<sup>-1</sup> in a nitrogen atmosphere. On ageing, peaks attributed to melting point (T<sub>m</sub>) and cold crystallisation temperature (T<sub>c</sub>) where amorphous regions rearranged into a crystalline phase. This was shown by the shift from two peaks at 121°C and 128°C to a single peak spanning 116–117°C. The shift can be attributed to a reduction in molecular weight and in flexibility of PLA due to hydrolytic degradation. A change in the number and dimensions of crystalline spherulites in PLA on degradation results in increased haziness of the material (Zhang et al. 2008).

Although Enviroware's fibre food box was expected to contain lignin fibres and adhesive in its construction, ATR-FTIR spectroscopy showed the presence of starch from C-H bands between 1100 and 1000 cm<sup>-1</sup>, but the absence of carbonyl groups that are characteristic of lignin and synthetic polymers. No changes in chemical structure were detected on ageing for 60 days.

Protein components of soya in bio-adhesive, reflected in N-H bands at 2800 cm<sup>-1</sup> underwent oxidation after 60 days at 70°C in both high and low RH environments. Humic acid lost bound water after 60 days at 70°C in dry conditions illustrated by broadening bands at 3500 to 3000 cm<sup>-1</sup> and generated acid groups by oxidation reflected in the intensification of bands at 1700 cm<sup>-1</sup>.

## **Evolution of acidic degradation products**

Acid-detection (A-D) strips, paper-based indicators that employ bromocresol green (3',3",5',5"-tetrabromo-m-cresol-sulfonephthalein) to detect and monitor the development of acids by photographic materials, were used to identify any acids developed by bioplastics (Image Permanence Institute 2001). When placed in a closed container with the sample material, the

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### CONCLUSION

Petroleum-based LDPE and HDPE and their bioplastic equivalents were chemically identical and exhibited similar degradation pathways and rates, substantiated by FTIR spectroscopy and TGA. Bioplastic polyethylenes can therefore replace directly traditional packaging materials. However, their limited availability in Europe and increased costs restrict their use today.

Polylactic acid-based food containers and bags were inferior to petroleumbased polyesters, including polyethylene terephthalate, used in conservation. They exhibited poor resistance to water, poor cohesion, loss of optical properties and production of volatile acids on ageing, particularly at high RH levels. Polylactic acid hydrolysed to form aldehydes, ketones and acids detected by thermal analysis and A-D strips. It would therefore not satisfy the ICOM code for materials used in museums. Food containers made from sugarcane and bamboo fibres were stable on ageing, although their lack of transparency limits their suitability as packaging for objects.

Soya adhesive formed strong, flexible and highly water-reversible bonds with wood, paper, glass, PVC and polyethylene, although its tendency to foam requires the use of antifoaming agents and ultrasound treatments during preparation. These properties suggest soya to show potential as a bio-adhesive for inorganic and organic museum objects including plastics, although its cream colour makes it unsuitable for white materials. Humic acid consolidant formed cohesive films which adhered well to wood, paper and soil. Although its brown colour limits its applications and its increasing acidity on ageing makes it unsuitable for long-term conservation, its high adhesive properties and reversibility in cold water suggest that it would find applications during archaeological excavations to temporarily secure loose sand and soil.

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### **MATERIALS LIST**

Biopolyethylenes (LDPE, HDPE) www.braskem.com.br/products

Chemicals from Sigma-Aldrich www.sigmaaldrich.com

Dura-Touch premium disposable vinyl gloves by Ansell www.ansell.com

High Tack Fish Glue from Conservation Resources www.conservationresources.com/Main/section\_34/section34\_18.htm

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# Melinex 401 polyester

http://libraryaccessories.com/conservation-preservation-materials.html

Microscope slides VWR https://dk.vwr.com/store/

Minigrip Plain Premium Red Line reseatable zipper bag http://products.minigrip.com/category/premium-red-line-reclosable-zipper-bags

Polylactic acid salad container (P700) and wood- fibre food container Enviroware Ltd, www.enviroware.co.uk/

Polylactic acid/cornstarch carrier bag from Joka, www.joka.dk

Polymethylmethacrylate (PMMA) sheets Rias A/S, www.rias.dk

Sobind Balance LVL soya powder G.B.C. (Speciality Chemicals) Ltd., Oxon, United Kingdom info@gbcspecs.co.uk

Tyvek UV Soft Structure – Style 1460 www.materialconcepts.com/tyvek/uv-soft/

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