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Identification of polymer-based artefacts from the former Wheeler Residence at the Mernda archaeological site in Victoria, Australia: A comparison of attenuated total reflectance and reflectance spectroscopic techniques

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Abstract
The purpose of this investigation was to identify the polymers in artefacts recovered from archaeological excavations at the former Wheeler Residence using attenuated total reflectance Fourier transform infrared and reflectance spectroscopy. The results from both techniques are compared and discussed. Identification will assist with the long-term management and storage of these polymer-based artefacts. Evaluation of these two sampling techniques included whether the physical characteristics and polymer type of the artefacts favoured the use of one technique over the other. The inherent challenges of archaeological artefacts such as cataloguing conventions, awkwardly shaped fragments and soil encrustations complicated the analysis. Of the 270 samples analysed, 67% were identified as containing ten different types of polymers, with the remaining 33% consisting

INTRODUCTION

Plastics are polymers or 'long chains of repeating molecules (monomers) (composed) predominantly of carbon and hydrogen atoms' (Mossman 1997, 1) which can be transformed through the application of heat or a solvent into a flexible shape via moulding or pressing (DuBois 1972, 1–2). The term 'polymer' is used herein to describe materials consistent with the above definition, including rubber and plastic. Polymer-based artefacts have been recovered from excavations and are thus represented in Australian archaeological assemblages. In particular, 255 finds consisting of 453 fragments were recovered from excavations at the Wheeler Residence in Mernda, Victoria. The site was the childhood home of Sir Kenneth Wheeler, a prominent Australian politician.

Fourier transform infrared (FTIR) spectroscopy is used extensively to characterise polymers (Scheirs 2000, 71) as is attenuated total reflectance (ATR) (Artioli 2010, 115, Nel et al. 2010). The advantage of the latter method is that no sample preparation is required, making the technique non-invasive and non-destructive. ATR has been used with a measure of success to identify adhesives on archaeological pottery (Nel et al. 2010, Noake et al. 2017) and historical polymers (Mitchell et al. 2013). The introduction of reflectance spectroscopy modules has enabled various external reflection sampling techniques (Fringeli 2000, 95) in which the sample needs only to be held in front of the module (Picollo et al. 2014, Saviello et al. 2016). Previous applications include the analysis of technological devices and contemporary artworks (Saviello et al. 2016). ATR and reflectance spectroscopy are ideal in the analysis of heritage polymers when destructive techniques are generally not permitted, the artefacts are fragile or in situ analysis is required.

The advantages of using ATR and reflectance spectroscopy in identifying the archaeological polymer-based artefacts in the Wheeler assemblage were:

1. These methods allowed the site to be dated, by matching archaeological polymers to the historical development of polymers common to particular eras, thus assisting with understanding the site's material culture, especially in the absence of a clear stratigraphy.
2. They contributed to determining the optimal storage of the artefacts. Described as 'malignant' by Williams (2002), cellulose nitrate (CN), cellulose acetate (CA), polyvinyl chloride (PVC), polyurethane (PU) and rubber can produce harmful substances that accelerate the deterioration

of unidentified polymers, encrusted polymers in which only soil bands or other materials such as glass were identified. Although reflectance achieved better results for certain types of artefacts, it also revealed limitations. The identified polymers are compatible with the proposed occupation of the site from 1852 to the 1970s.

of these polymers (Kenegan and Quye 1999) and affect the condition of neighbouring materials. Identification informs the housing of polymer artefacts in an appropriate storage environment. Degradation factors such as ultraviolet radiation, oxygen, water and/or corrosive degradation products can be minimised and thus improve long-term preservation via storage in an environment that is dark, sealed, has a low relative humidity (RH) and/or is well ventilated.

HISTORICAL BACKGROUND

Modern-day Mernda is a north-eastern suburb of Melbourne in the state of Victoria, Australia. Due to plans for a large residential development project, archaeological excavations were conducted in 2015–16 at 110 Sackville Street. The study area measured approximately 10.47 hectares, however archaeological excavations focused on 450 m^2 of land located on the eastern border of the study area (Mitchell 2018, 1) (Figure 1). The site dates from approximately 1852 to the 1970s (Figure 2), although post-1970s modern debris may have contributed to the site before excavations commenced in 2015. The archaeological site is significant due to its association with Sir Kenneth Wheeler (1912–1996) (Mitchell 2018), a member of the Victorian Parliament and its Speaker from 1973 until 1979 (Parliament of Victoria 2003). Consequently, the former Wheeler Residence is a Heritage Inventory site (no. H7922-0491) protected under the Victorian Heritage Act 1995, later re-enacted by the Heritage Act 2017 (Heritage Council of Victoria 2018).

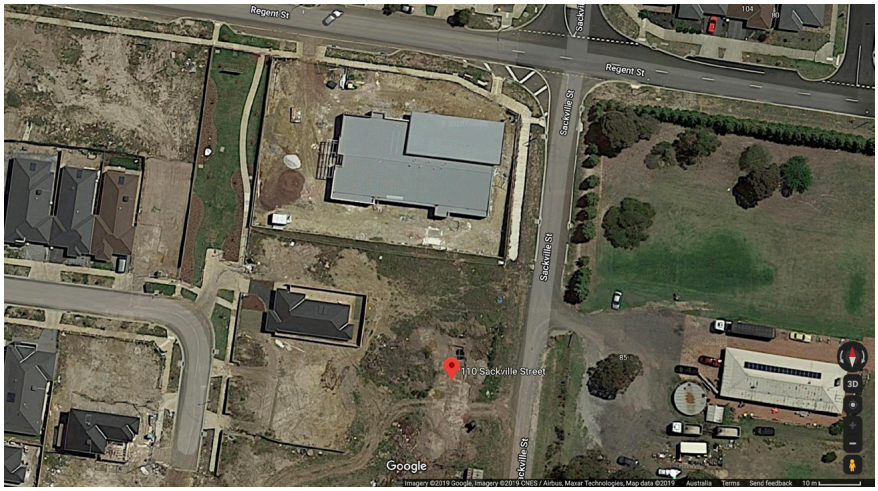

Figure 1. Study area and site (Google Maps 2019)

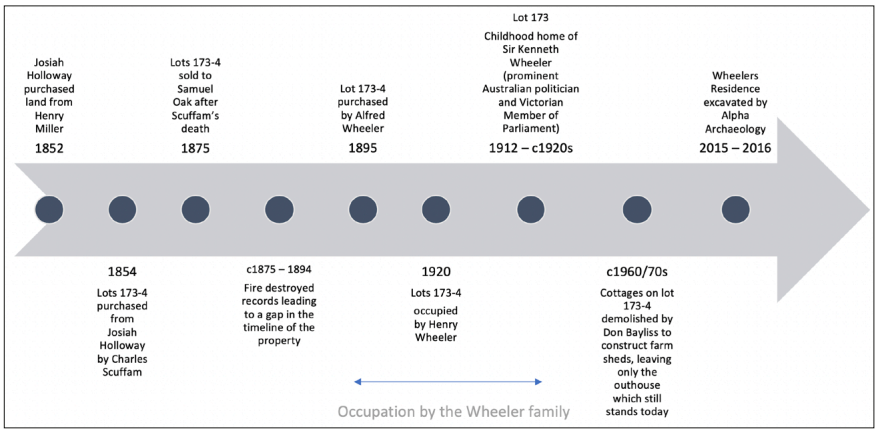

Figure 2. Timeline of the occupation of the Wheeler Residence site

POPULARITY OF POLYMERS

The 19th century saw the invention and development of vulcanised rubber (VR) and semi-synthetic polymers (Slack 2002, Shashoua 2008). This period of innovation continued until the early years of the 20th century leading up to World War I, when fully synthetic polymers such as Bakelite became commercialised (Rasmussen 2018). The end of World War II signalled the start of the plastics revolution whereby product diversity was spearheaded by post-war innovation and scientific progress (Todd 2007, 83).

The advantages of polymers as an alternative to traditional materials explain their popular use throughout the 20th century and into the present. Polymer manufacture grew exponentially in Australia during the 1950s and 1960s (Karpfen and Nagarajan 1988, 8), with companies such as Nylex (Moulded Products (Australasia) Ltd) (Museums Victoria 2019), BASF Australia and Monsanto Australia Limited (Karpfen and Nagarajan 1988, 10–11) manufacturing the most common thermoplastics: low-density polyethylene (LDPE), high-density polyethylene (HDPE), polypropylene (PP), polystyrene (PS), PVC, acrylonitrile butadiene styrene (ABS), acrylics and polyvinyl acetate (PVAc). A brief timeline of the development and popular use of the polymers identified in this paper is provided in Figure 3.

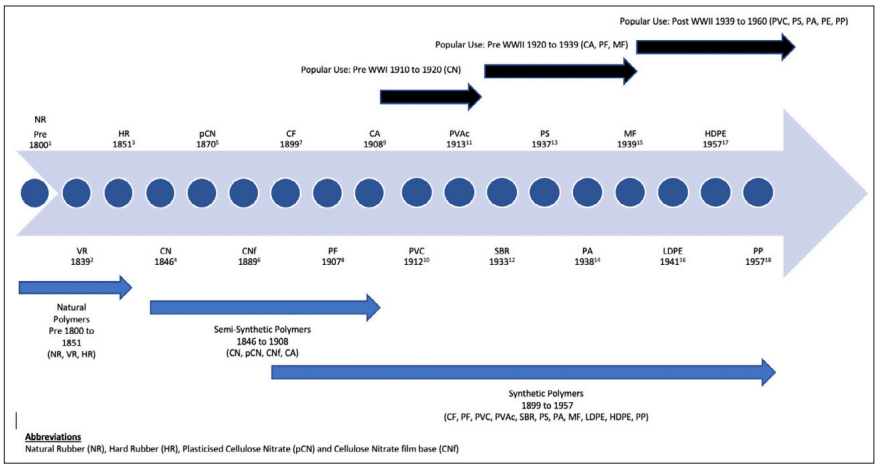

Figure 3. Timeline highlighting the development of the types of polymers found in the Wheeler assemblage: 1 to 10 and 13 to 18 (Shashoua 2008), 12 (Kauffman 2016), 11 (Zecchina and Califano 2017)

MATERIALS AND METHODS

Investigations involved categorisation, non-invasive examination and photographic documentation, and FTIR analysis.

Categorisation

The 255 artefacts, comprising 453 fragments, were separated into 35 categories according to their physical description (Figure 4), which allowed comparisons of artefacts with similar appearance or function.

Non-invasive examination

The non-invasive examination of the 255 artefacts involved condition reporting and photographic documentation using a Nikon D5300 digital single-lens reflex (DSLR) camera with a Nikon AF-S DX Nikkor 18–55 mm f/3.5–5.6G VR II zoom lens.

Figure 4. Polymeric artefacts in the Wheeler assemblage. Photographs: Clare Kim, March 2018

Multiple fragments

Multiple fragments are accessioned under the same number. To assist with identification, one sample was obtained from the most representative fragment of an accessioned artefact if the materials appeared to be identical. For example, if an accessioned artefact consisted of three pieces of identical material, then the least degraded fragment was selected. An exception to this rule was when the least degraded fragment was curved or awkwardly shaped, making it unsuitable for FTIR analysis. In addition, if the fragments differed in their appearance, each fragment under the same accession number was sampled. Lastly, if an accessioned artefact consisted of two or more distinct parts, each part was sampled.

ATR data acquisition and analysis

Data acquisition was adapted from the methodology used to identify adhesives on archaeological ceramics (Nel et al. 2010): IR absorbance spectra were collected using a Bruker Alpha-P FTIR spectrometer equipped with a diamond ATR window (pre-cleaned using isopropanol) in the spectral range of 4,000–375 cm^{-1}, with 32 co-added scans (sample and background scan times) at a spectral resolution of 4 cm^{-1}. The spectrometer weighs only 7 kg (Bruker 2011) and occupies a space the size of an A4 sheet of paper (Nel et al. 2010, 65) and is thus portable. Each artefact was either clamped, with the object's surface protected with a piece of card (if sturdy), partially clamped (if fragile) or manually pressed against the diamond window (if awkwardly shaped and/or badly degraded).

Data analysis similar to that described by Nel et al. (2010) involved the use of OPUS software (version 7.5) to collect and analyse IR spectra from artefacts. The data were then matched against a commercial spectral library and an in-house database of reference spectra acquired from a control set of authentic plastic samples via the Quick Compare function. Identification was visually confirmed by overlaying each unknown IR spectrum with that of a known reference sample and visually matching characteristic peaks.

ATR spectra of references were obtained from the following sources:

- samples provided at Thea van Oosten's August 2005 *Working with Plastics* workshop and Yvonne Shashoua's July 2015 *Conservation of Plastics* workshop, both delivered in Melbourne, Australia;
- objects collected as part of the in-house collection; and
- polymers in the ResinKit manufactured by the Resin Kit Company (Rhode Island, United States) and purchased in 2017.

Comparisons were also made with the published ATR spectra of known polymers (Scheirs 2000, Socrates 2001, Shashoua 2008, Saviello et al. 2016) and the IRUG database (Price et al. 2007).

Reflectance data acquisition and analysis

Artefacts whose ATR spectra could not be obtained were analysed using reflectance spectroscopy to determine whether the method would aid in polymer identification.

Reflectance spectra were acquired using an external reflection (ER) module attached to the spectrometer described above. The spectral range and resolution were the same as utilised in the ATR analysis. Depending on the size of the artefact, a small (3.75 mm diameter) or medium-sized (5.75 mm diameter) sampling window was used to generate spectra with either 64 or 32 co-added scans, respectively, within a 15 mm working distance. A gold mirror attachment was used to obtain background measurements with 64 or 32 co-added scans. Images of the artefact were captured on a Samsung Galaxy Note 9 and the integrated video camera (IC Capture software) attached to the ER module. Kramers-Kronig transformation (KKT) was applied to the reflectance spectra to remove distortions and convert them into absorbance spectra to facilitate interpretations of key functional groups (Fringeli 2000).

Comparisons were made with an in-house database of reflectance spectra. Identity was visually confirmed by overlaying unknown reflectance spectra with the spectra of known references and visually matching characteristic peaks. If required, the transformed KKT absorbance spectra were also compared with ATR reference spectra as an additional confirmation tool.

RESULTS AND DISCUSSION

ATR analysis

Of the 270 samples analysed, 56.3% were identified by ATR analysis as styrene-butadiene rubber (SBR), CN, CA, plasticised PVC (pPVC), unplasticised PVC (uPVC), PS, casein formaldehyde (CF), phenol formaldehyde (PF), melamine formaldehyde (MF), polyamide (PA), polyethylene (PE) and polypropylene (PP). The remaining 43.7% consisted of either plastics that were unidentified, artefacts in which only soil was measured or other materials such as glass. The ATR reference spectra are provided in Figure 5.

Polymers with a soft, flexible and/or flat surface could be identified more readily as they could be clamped. Although most of the spectra were of

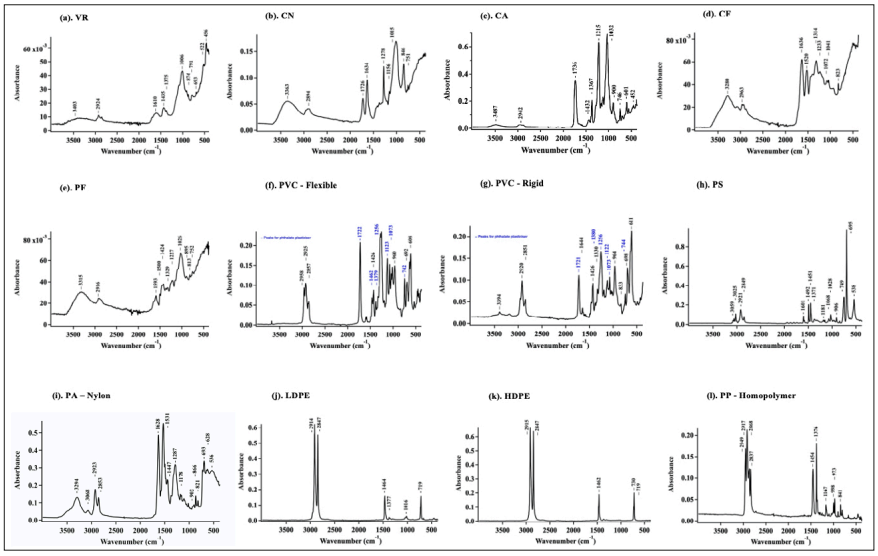

Figure 5. ATR spectra of known reference samples: (a) degraded VR sample from an archaeological artefact; (b) CN sample from Yvonne Shashoua's July 2015 *Conservation of Plastics* workshop; (c) resin kit no. 11 CA; (d) CF sample from Yvonne Shashoua's July 2015 *Conservation of Plastics* workshop; (e) PF sample from Thea van Oosten's August 2005 *Working with Plastics* workshop; (f) resin kit no. 29 PVC flexible; (g) resin kit no. 30 PVC rigid; (h) resin kit no. 1 PS – general; (i) resin kit no. 14 PA – nylon; (j) resin kit no. 24 LDPE; (k) resin kit no. 25 HDPE; (l) resin kit no. 27 PP – homopolymer

good quality, it was often difficult to assign peaks due to the additional presence of plasticisers, soil contamination and artefact degradation. As the characteristic bands for LDPE (1,380, 1,365 cm^{-1}) (Socrates 2001) tend not to overlap with soil peaks, they were easier to identify. However, the identification of some categories of objects proved to be problematic. Small spherical shapes made it difficult for beads or buttons to be clamped securely for analysis, resulting in low-quality IR spectra. Silica bands were observed for glass artefacts at 910–1,034 cm^{-1}.

Reflectance analysis

Reflectance analysis and comparisons with references improved the identification process (Figures 6, 7). Artefacts that could not be identified

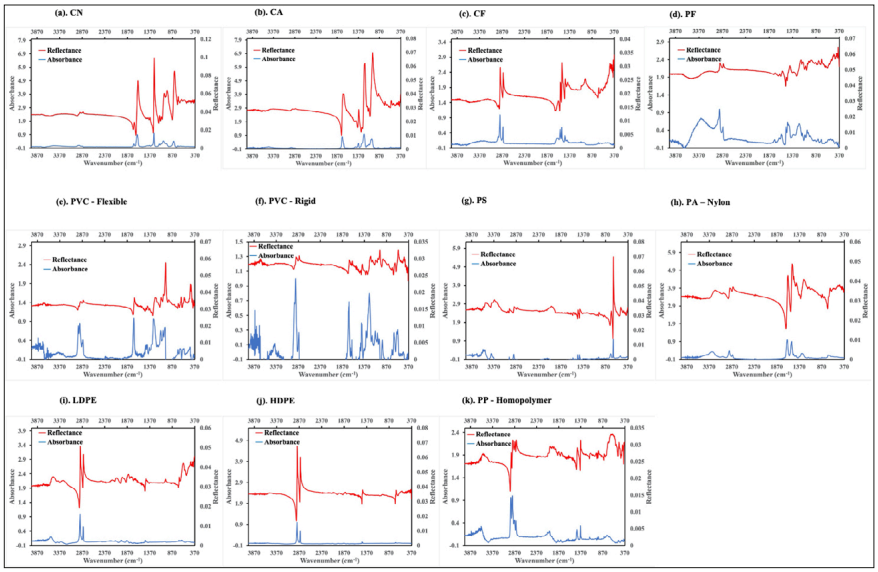

Figure 6. Reflectance and calculated absorbance spectra (KKT) of known reference samples: (a) CN sample purchased from eBay; (b) resin kit no. 11 CA; (c) CF sample purchased from GPS Agencies; (d) PF sample purchased from eBay; (e) resin kit no. 29 PVC flexible; (f) resin kit no. 30 PVC rigid; (g) resin kit no. 1 PS - general; (h) resin kit no. 14 PA – nylon; (i) resin kit no. 24 LDPE; (j) resin kit no. 25 HDPE; and (k) resin kit no. 27 PP – homopolymer

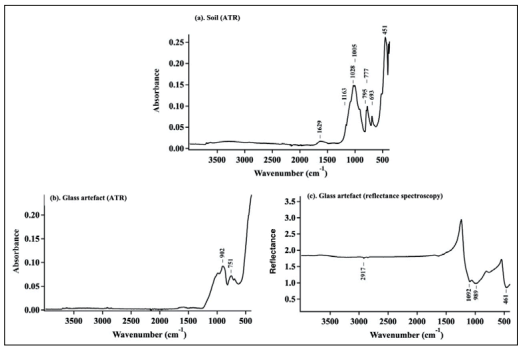

Figure 7. ATR spectra of (a) soil sample from WHE 03039 and (b) degraded glass artefact. Reflectance spectrum of (c) degraded glass artefact taken in reflectance mode

using ATR due to their hardness or uneven forms, which prevented them from being held steadily in the clamp, could be identified using reflectance analysis. For example, WHE 03079, a green plastic pig, was identified as PS whereas the ATR spectrum of the same artefact was inconclusive (Figure 8). A comparison between the ATR, reflectance and calculated absorbance spectra of PVC demonstrated that ATR is the most reliable method of interpretation when the artefact also contains plasticisers. Due to their degradation, the plasticiser peaks tended to occur at lower intensities, indicative of plasticiser loss from the degrading artefact.

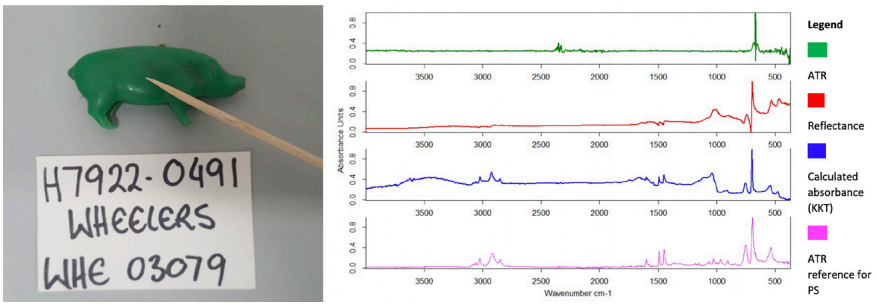

Figure 8. Comparison of the ATR, reflectance, calculated absorbance (KKT) and ATR reference spectra of a PS artefact

Reflectance also aids in the identification of polymers previously misidentified using ATR (SBR and MF) and is more suited to analysing thick, hard and rigid polymers. The characteristic bands for certain types of materials (formaldehydes, VR/vulcanite and glass) appear more distinct than in their ATR spectra. The sample spectra were overlaid with the spectra of known samples of degraded vulcanite, as the spectra for newly manufactured VR are significantly different by comparison. This analysis helped to identify many artefacts initially mislabelled as 'Bakelite' but subsequently determined to be VR. Accurate identification should result in correct classification, labelling and storage.

Overall results

The results demonstrated the presence in the assemblage of rubber (VR), semi-synthetic polymers (CN and CA), synthetic polymers (formaldehydes, PVC, PS, PA, PE and PP) and other materials (glass, soil) (Table 1). Although the number of artefacts identified increased from 56.3% to 67%, many polymers could not be identified due to the limitations described below.

LIMITATIONS

Polymer shapes

Concave structures were easier to analyse than convex, bulbous shapes, which could not be clamped. Balls, for instance, could not be clamped, as the ATR spectrometer has a limited height adjustment capacity and these shapes did not fall within that range. Bottle caps could not be analysed properly as the clamp was too big to fit inside the caps to hold them securely. Generally, long and narrow shapes such as sticks were the most amenable to ATR analysis since they could be securely clamped.

Soil encrustations

All artefacts had various degrees of soil encrustations. The soil sample from the WHE 03039 blue plastic fragment showed characteristic bands due to silica and clays in the fingerprint region below 1,050 cm^{-1} (Yeasmin et al. 2017) (Figure 7). Unknown polymeric materials were most easily identified based on comparisons of their spectra with those of known pure polymers (Laganà and Keneghan 2012, 37). Dominant soil bands

Table 1. Artefact categories and types of polymers identified in the Wheeler assemblage using ATR and reflectance spectroscopy

		Rubber	Semi-synthetic polymers		Synthetic polymers									Other		Unidentified	Total	Original
No.	Categories	VR	CN	CA	CF	PF	pPVC	uPVC	PS	PA	LDPE	HDPE	PP	Glass	Soil	Unknown		
1	Animal-shaped			1					2								3	3
2	Ball															2	2	2
3	Bead													31			31	30
4	Button	4	2		1	5								7	1		20	20
5	Cap	4				1									1		6	6
6	Comb	3	6						10								19	14
7	Embellishment		1			1								1			3	3
8	Film		1												9		10	10
9	Fragments	4	1			3		1	51			1	3	2	3	2	71	69
10	Hair clasp		1														1	1
11	Handle								2							1	3	3
12	Knot														1		1	1
13	Lid					1				1	1		1				4	4
14	Mallet															1	1	1
15	Miscellaneous	7	1			1			1			1	1		3	9	24	19
16	Moulded								6			1				1	8	8
17	Peg								1								1	1
18	Pipe							1									1	1
19	Plate	1															1	1
20	Plug	1															1	1
21	Rectangular	2															2	2
22	Ring														4	1	5	5
23	Rope														1		1	1
24	Seal	1															1	1
25	Sheet														1		1	1
26	Spoon								1								1	1
27	Stationery	1							2								3	3
28	Stick								4								4	4
29	Stopper	2				1										2	5	5
30	Strip						2		1							1	4	4
31	Toothbrush		1														1	1
32	Toy			1		1			3		6				1		12	12
33	Tube	3				1			2							2	8	8
34	Washer	1															1	1
35	Wire						8				1					1	10	8
Total sampled		34	14	2	1	15	10	2	86	1	8	3	5	41	25	23	270	255
% of 270 samples		12.6	5.2	0.7	0.4	5.6	3.7	0.7	31.9	0.4	3.0	1.1	1.9	15.2	9.3	8.5		

Notes:

The total number of samples identified exceeds the total number of accessioned artefacts for that category. This is due to various fragments belonging to one accessioned artefact being composed of different materials. Hence, each component was analysed, thereby increasing the total number of samples analysed.

prevented polymer identification for some artefacts. Cleaning was not an option due to the fragility and contextual importance of the debris.

Styrenic differentiation

Differentiation between pure PS, high-impact polystyrene (HIPS), styrene acrylonitrile (SAN) and ABS is possible based on the presence of the butyl rubber peak at 960 cm^{-1} and/or the acrylonitrile peak at 2,200 cm^{-1} (Scheirs 2000). Unfortunately, noise and soil impurities in the IR spectrum made it difficult to identify these subtle differences.

Additives and plasticisers

There are multiple potential additives in PVC (Socrates 2001, 269). As plasticisers tend to migrate to the surface of an artefact as it deteriorates, it was easier to identify PVC based on the presence of peaks associated with common plasticisers.

Degraded elastomers

Factors relating to the deterioration of elastomers further complicated the analysis, as degraded rubber exists in many forms depending on the catalyst for degradation (Bin Samsuri 2010). Rubber artefacts were often covered in soil and were too degraded to allow their FTIR analysis.

IMPACT ON CONSERVATION AND COLLECTION MANAGEMENT

Polymer degradation

As the majority of the deterioration in polymers is physical, chemical and/or biological in nature (Shashoua 2008, 153), it is important to understand the mechanisms of degradation to devise mitigation measures (Table 2). All artefacts in this assemblage exhibited various degrees of mechanical damage. Malignant polymers (VR, CN, CA and PVC) accounted for

Table 2. Degradation of polymeric archaeological artefacts

Polymer	Physical			Chemical			
	Mechanical	Interaction	Migration of additives	Ultraviolet (UV) radiation and light	Heat	Oxygen	Water
VR	✓			✓	✓	❍	
CN	✓		✓	✓	✓	●	✓
CA	✓			✓	✓	●	✓
pPVC	✓	✧	✓	✓	✓	●❍	✓
uPVC	✓	✧	✓	✓	✓	●❍	
PS	✓			✓	✓	●	
CF	✓			✓	✓	●	✓
PF	✓			✓		●❍	
PA	✓			✓	✓	●	✓
PE (HD/LD)	✓	❑✧		✓	✓	●❍	
PP	✓	❑✧		✓	✓	●❍	

Notes (Shashoua 2008):
✓ Degradation observed
❑ Discolouration/tackiness
✧ Expansion or contraction
● Photooxidation
❍ Thermal-oxidation

23% of the artefacts' constituents. Given the volatility and propensity of malignant polymers to affect other artefacts, their presence warrants preventive action to inhibit further deterioration.

Informed decision-making

Identifying the polymer type is the first step towards the conservation and management of archaeological polymer-based assemblages. Understanding how polymers degrade assists in the determination of appropriate conservation measures. Such decisions may include isolating malignant polymers, creating appropriate storage, prioritising treatment and duplicating or de-accessioning degraded artefacts.

Storage requirements

All artefacts in the Wheeler assemblage are currently stored in PE ziplock bags that create a microenvironment around each item. PE is stained when in close contact with VR (as described in Shashoua 2008, 153). PE also encourages the absorption of plasticisers used in PVC, exacerbating the latter's degradation (Shashoua 2001, 97). Alternative enclosures such as archival corrugated board or glass may be more appropriate for the storage of some polymers.

A host of polymers can undergo degradation when exposed to environmental factors such as fluctuations in temperature, RH, light and pollution (Pastorelli et al. 2014). The method of deterioration can be used to select the storage option most appropriate to slow its continuation (Table 3). For example, CN deteriorates through photooxidation (Berthumeyrie et al. 2014) and generates acid when it undergoes hydrolysis (Quye et al. 2011, 1370). Collection manuals advise that CN be stored in a well-ventilated fireproof area with appropriate absorbers to remove any noxious vapours that are released and to prevent the build-up of such gases (Williams 1994, Godfrey

Table 3. Storage recommendations for polymeric archaeological artefacts

Polymer	Storage				
	Dark	Temperature 18°C ± 2[1]	RH 55% ± 3[1]	Enclosed	Ventilated
VR	✓	✓	✓		✓✱
CN	✓	✓	♦		✓⅄•✱
CA	✓	✓	♦		✓⅄•✱
pPVC	✓	✓	♦	✓O	
uPVC	✓	✓	✓	✓O	
PS	✓	✓	✓	✓	
CF	✓	✓	!	✓⅄O	
PF	✓	✓	✓	✓O	
PA	✓	✓	♦	✓⅄	
PE	✓	✓	✓	✓O	
PP	✓	✓	✓	✓O	

Notes:
[1] Shashoua 2008
♦ 30% RH (Lovett and Eastop 2004)
! 60% RH (Godfrey 2017)
⅄ Silica gel
• Zeolites
O Oxygen absorbers (Lambert et al. 1992, Grattan and Gilberg 1994)
✱ Isolate

2017). However, many archaeological assemblages in Australia are kept on premises without environmental controls. Therefore, ventilation may increase deterioration if the RH is high. Consequently, the most appropriate storage options for polymers in archaeological collections will depend on the final storage repository and the means to procure appropriate storage materials and enforce environmental parameters.

FUTURE RESEARCH

For degraded rubbers, IR analysis is problematic. Photoacoustic spectroscopy (PAS) can analyse solid samples when other techniques are ineffective (Rosencwaig 1980, Khandpur 2006) and FTIR–PAS has been applied in the analysis of polymers (McClelland et al. 2003, Zhang and Urban 2004). The characterisation and analysis of natural rubber and VR (Hendra and Jackson 1994, Xue 1997, Taksapattanakul et al. 2017, Bokobza 2019) indicate that Raman spectroscopy may also be an effective alternative.

CONCLUSION

A non-invasive visual examination of the artefacts from the Wheeler assemblage was unable to positively identify their polymer types, although it provided useful supplementary information as did the condition reports cataloguing the physical characteristics associated with polymer degradation. However, ATR and reflectance spectroscopy successfully identified the materials and confirmed the dating of the site. Moreover, both methods helped to inform conservation decisions. FTIR identified 247 of the 270 samples analysed, of which 181 were composed of polymers, 41 of glass and 25 of soil. Nonetheless, several limitations complicated the identification process such that 23 samples could not be identified. The range of excavated polymers matched the timeline of the site's occupation. The findings may assist with determining the storage of other polymer-based archaeological assemblages, such as the need to enclose or ventilate a polymer within a PE bag or to replace PE bags with more appropriate housing materials.

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