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Benign solvents for recycling and re-use of a multi-layer battery pouch†

Jean E. Marshall,^{id}*^a Bethany Middleton,^a Dominika Gastol,^{id}^b
Roberto Sommerville,^{id}^b Con R. McElroy,^{id}^c Emma Kendrick^{id}^b and
Vannessa Goodship^a

This article describes a process for the repair and re-use of an aluminium-containing pouch used as an outer casing for a Lithium-ion battery cell. As Lithium-ion batteries become more widespread, particularly with their increasing use in the automotive industry and in consumer electronics, recycling them is becoming an important challenge. Current recycling approaches for Li-ion batteries focus on reclamation of the high-value metals found in the electrodes. However, in order to minimise the environmental impact of the battery it would be optimal to be able to reclaim and re-use other components. Since many battery cells consist of an electrode stack held inside an outer pouch, we herein describe the structure of such a pouch and suggest methods for selectively stripping and repairing the inner layer, allowing the material to be re-used. We investigated the use of three different solvents (xylene, limonene, and 2,2,5,5-tetramethyloxolane) for the selective stripping of the polypropylene (PP) layer on the inner side of a pouch material. Each solvent was tested on both 'pristine' pouch material (as obtained from the manufacturer) and pouch material that had previously been part of a battery. For 'used' pouch material, the overall thickness of the film decreased from 150–160 μm to 70–80 μm in under 1 h for all three solvents (corresponding to the removal of the PP layer), whereas for the 'pristine' pouch higher temperatures and longer times were required; this result is suggestive of some polymer degradation during the life of the pouch. Following the removal of the PP layer, virgin PP was subsequently added to renew the multi-layer structure.

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1 Introduction

A recent report by the International Energy Agency (IEA) demonstrates the extent to which electric vehicles are increasingly attractive to consumers,¹ stating that "There were 10 million electric cars on the world's roads at the end of 2020, following a decade of rapid growth. Electric car registrations increased by 41% in 2020, despite the pandemic-related worldwide downturn in car sales in which global car sales dropped 16%". Battery Electric Vehicles (BEVs) differ from fossil fuel-powered cars in that they contain electric motors instead of internal combustion engines, and are powered by battery packs. Electric vehicles (EVs) have numerous environmental advantages over traditional cars – they do not emit exhaust fumes from a tailpipe, and their batteries can be recharged using energy

generated from renewable sources. However, there is still a large environmental cost associated with the manufacture of batteries for EVs,^{2–5} and therefore as the number of EVs on the road increases it is becoming a matter of pressing concern to recycle battery packs as they come to their end-of-life.⁶

Recycling Li-ion batteries poses some difficult challenges – there are several different chemical components present in each battery, and separating each of these out to make pure waste streams can be a complex process.^{7,8} The majority of automotive battery packs are based on lithium-ion chemistry,^{9–11} with a lithium-containing metal oxide at the cathode and graphite at the anode. Furthermore, the design of an electrical cell can take different formats. For electric vehicles, there are 3 main formats in use – pouch cells, cylindrical cells and prismatic cells.¹² According to a recent estimate,¹³ approximately 40% of cells produced in passenger vehicles are based on the pouch format. Any recycling process for these electrical cells will need to be tailored to specific cell chemistry and format. Furthermore, the toxicity and flammability of some of these chemical components can impose strict requirements on the facilities needed to safely carry out a recycling procedure. Despite these issues, it is vital that effective recycling processes

^a WMG, University of Warwick, Coventry, CV4 7AL, UK.

E-mail: Jean.Marshall@warwick.ac.uk

^b School of Metallurgy and Materials, University of Birmingham, Edgbaston, Birmingham, B15 2TT, UK^c Department of Chemistry, University of York, Heslington, York, YO10 5DD, UK† Electronic supplementary information (ESI) available: Hansen solubility simulations for PP in solvents. See DOI: <https://doi.org/10.1039/d2ma00239f>

pouch, with a discussion of how used materials can be repaired or recycled. Fig. 1 shows photographs of a cell taken out of a module, followed by a diagram depicting the contents of the electrode stack inside the cell. The electrode stack contains layers of aluminium foil, copper foil, cathode/anode material ('black mass') and a separator; all of these materials are infused with an electrolyte which contains a lithium salt, and care must be taken during disassembly due to the flammable nature of this electrolyte.

A patent for a commercial pouch material²⁶ describes a process in which an aluminium foil is coated in adhesive then polymer is attached to each side of the foil. This gives rise to a layered structure, as shown in Fig. 2(a). A cross-section of our pouch material (D-EL408PH(3), manufactured by Dai Nippon Printing Co.) was examined by SEM, and shown to also have a layer structure, as shown in Fig. 2(b), and therefore we suggest that our pouch was manufactured by a similar process.

At the time of writing, the cost of the pouch material is 77 SGD (42 GBP) per m², which we estimate to be 190 GBP per kg, while both aluminium and polypropylene cost less than 1 GBP per kg.^{27,28} Therefore, it is clear that a substantial part of the value of the pouch is in its design and manufacture; being able to re-use the pouch material is potentially more cost-effective than being able to reclaim the raw materials from it. Metal-containing pouches are used in many industries other than batteries; for example, in food/pharmaceutical industries where effective barrier materials are required to protect the contents of the pouch from ambient conditions. Batteries contain flammable and toxic materials, so reclaimed battery pouches are unlikely to be re-used in food contact or medical applications;

however, lessons learned from attempts to reclaim battery pouches could be applied to reclaiming used pouch material from these other sectors.

An optimal reclamation route for the pouch material would involve being able to clean and repair it so that it can be re-used. Any contaminants associated with the other components of the battery (such as the electrolyte) will principally be found on the 'inner' side of the pouch (adjacent to the electrode stack), therefore it would be advantageous to be able to selectively strip the inner polymer layer (while leaving the outer layer intact) and re-coat the inside of the pouch. In this manner, the economic value of the pouch material can be retained while it is made ready for re-use. We herein present a characterisation study of a commercial pouch material, followed by solvent extraction to selectively strip the inner layer from the pouch, followed by the addition of a new polymer layer to the inner side of the pouch. Previous studies by other authors have indicated that solvent stripping methods can be favourable, in terms of energy cost, compared to other recycling approaches.^{29,30} As part of our study, we investigate two relatively 'benign' solvents, limonene and TMO, and show that these solvents can selectively strip the inner layer allowing the pouch to be repaired.

2 Results and discussion

As mentioned above, this study was carried out using pouch material D-EL408PH(3), manufactured by Dai Nippon Printing Co. The Material Safety Data sheet for this pouch material gives the approximate composition of the material, as shown in Table 1.

This composition is compatible with an aluminium foil layer coated on both sides with polymer. We investigated 'pristine' pouch material (as received from the manufacturer) and compared this to 'End of Life (EoL)' pouch material (from a battery pack at the end of life).

Fig. 3 shows dynamic scanning calorimetry (DSC) data to compare a 'pristine' pouch to a pouch at its EoL. For the 'pristine' (as-received from the manufacturer) pouch, signals are clearly seen at 140, 160, 220, and 260 °C; these correspond to polypropylene, acid-modified polypropylene, nylon, and polyethylene terephthalate, respectively. Interestingly, however, for the EoL pouch the signal at 160 °C has disappeared completely, while the signal at 140 °C has increased in intensity. Since polypropylene does not bond well to many substrates, we assume that the acid-modified polypropylene is included to ensure good adhesion between polypropylene and aluminium;



Fig. 2 (a) Schematic diagram of a layered pouch film (b) SEM image of a cross section through a pouch film, showing the layered structure.

Table 1 Composition of pouch. Data obtained from the material safety data sheet for this pouch material

Material	%	Melting point (°C)
Polyethylene terephthalate (PET)	8 ± 0.8	260
Nylon	8 ± 0.8	220
Adhesive	3 ± 0.3	
Aluminium	49 ± 4.9	650–660
Acid-modified polypropylene	16 ± 1.6	160
Polypropylene (PP)	16 ± 1.6	140



Table 2 Properties of 3 solvents for PP

Solvent	Boiling point (°C)	Hazards	
Xylene (mix of isomers)	136–140	H226	Flammable liquid and vapor
		H304	May be fatal if swallowed and enters airways
		H312 + H332	Harmful in contact with skin or if inhaled
		H315	Causes skin irritation
		H319	Causes serious eye irritation
		H335	May cause respiratory irritation
		H373	May cause damage to organs (hearing organs) through prolonged or repeated exposure
		H373	May cause damage to organs (central nervous system, liver, kidney) through prolonged or repeated exposure if inhaled
Limonene (mix of isomers)	170–180	H412	Harmful to aquatic life with long lasting effects
		H226	Flammable liquid and vapor
		H315	Causes skin irritation
		H317	May cause an allergic skin reaction
		H410	Very toxic to aquatic life with long lasting effects
TMO	112	H225	Highly flammable liquid and vapor
		H302	Harmful if swallowed

of solvents with low hydrogen bonding ability, low to moderate polarisability, moderate to high dispersivity. The three proposed solvents lie within this grouping, again suggesting good performance. Finally including solvents to which PP shows excellent resistance gave a broad covering of HSP space although with moderate to high hydrogen bonding ability being almost ubiquitous.

In order to test the response of the pouch material to the solvents chosen, pieces of pouch material were immersed in these 3 solvents at different temperatures and times, before being removed from the solvent bath, rinsed and dried. The thickness of the film was measured for each sample after drying, and these film thicknesses are collated in Fig. 5. There are clear trends in these data. When xylene is the solvent, at 90 °C, EoL pouches decrease in thickness to an eventual value of 70–80 µm within 1 h, and after this the thickness stabilises, presumably as no further PP is stripped from the surface. In the case of the 'pristine' pouch, however, this process still occurs rapidly at 120 °C but at 90 °C the process occurs more slowly and it appears that not all of the PP layer is stripped after 1 h. The fact that PP is stripped more readily from EoL pouches than for the pristine pouch is strongly suggestive of a chemical change occurring in the inner pouch layer during the battery's use; this finding is consistent with the DSC and FTIR data. Similar trends are found for TMO and limonene, with the EoL pouch being stripped more quickly than the pristine pouch. From these data, limonene is a particularly suitable replacement for xylene, stripping PP from the EoL pouch in 10–20 min at 90 °C. Fig. 6 shows the FTIR data obtained on the inner and outer surfaces after extraction in the 3 solvents; as expected, the polymer peaks on the inner surface are gone or dramatically reduced after solvent treatment, while the polymer peaks on the outer surface remain.

During manufacture of this type of pouch, the edges are bonded together by being compressed under heating, to form the pouch structure. As may be appreciated from the schematic diagram in Fig. 1(e), when the pouch is opened (to remove the electrode stack for recycling) by an incision made with a blade, the edges will remain bonded together. For the purpose of recycling the pouch, it would be advantageous to be able to

re-use the edge pieces along with the rest of the pouch material. We therefore also immersed an edge piece of the material in xylene in order to see whether the edges could be separated straightforwardly. As shown in Fig. 7(a and b), separation occurs readily during this soaking process. During a disassembly of the battery, using an approach of the type described in our previous work,²⁵ an incision will be made into the pouch in order to disassemble it, and therefore recycled pieces will be smaller and will need to be repurposed for smaller pouch cells. However, if the incisions required are kept to the minimum necessary to extract the cell contents, any bonded edges can be soaked in solvent for material recovery.

Fig. 7(c) shows a piece of inner pouch that has been only partially stripped with solvent; the inner surface appears dull and inhomogeneous. After being fully stripped, however, the surface becomes shiny again as shown in Fig. 7(d).

In order to further investigate the reusability of these pouch materials, an aluminium surface that had been stripped with solvent was covered with a new PP film, then compressed and heated to 160 °C so that the PP film softened and adhered to the Al surface, as shown in Fig. 7(e). Adhesion was tested by a simple cross-hatch test; the PP film was scored in a cross-hatch pattern as shown in Fig. 7(f) before being covered with duct tape. When the tape was pulled off the surface, none of the PP layer was removed. The adhesion of the 'new' PP film to the aluminium surface is further investigated in the SEM studies below.

As well as the aluminium layer, it is possible to recover the PP that has been stripped from the surface during solvent treatment. A vessel containing limonene, that had been used to strip the PP layer from of pouch material, was cooled to room temperature; acetone (a non-solvent for PP) was added and the PP readily precipitated out as a white solid. As shown in Fig. 7(g), this white solid has an identical appearance when recovered from pristine and EoL pouches.

We also carried out SEM-EDS analysis in order to further confirm the findings of this study. Fig. 8(a) shows elemental maps of a cross-section through a 'pristine' pouch. A thin Al film is clearly shown in the centre of the cross section, with carbon layers on either side due to the polymer layers; one





Fig. 5 Graphs to show thickness decrease with time of pouch samples immersed in solvents. Each data point represents an average thickness: 2 pieces of pouch were immersed in the solvent for the given time, and 3 thickness measurements were taken from each piece. (a) pouch samples from pristine and EoL pouch, immersed in xylene at 90 °C and 120 °C (b) Pristine and EoL pouch immersed in limonene at 90 °C. (c) Pristine and EoL pouch immersed in TMO at 90 °C.

of those carbon layers is more oxygen-rich than the other, presumably due to the polyester structure of PET in the outer layer (compared with the inner layer, which is PP and therefore expected to contain largely carbon and hydrogen only).



Fig. 6 FT-IR spectra of (a) the inner surface of an EoL pouch without treatment, and the inner surface of the pouch after treatment with solvent (b) the outer surface of an EoL pouch without treatment, and the outer surface of the pouch after treatment with solvent.

The same analysis was carried out on 'pristine' pouch that had been immersed in xylene at 120 °C for 2 h, and the results are shown in Fig. 8(b); the Al layer is intact as expected while the 'carbon' layer has been stripped from one side of the pouch. The remaining carbon layer is the 'oxygen-rich' layer, *i.e.* the PET-containing outer layer. These images corroborate our finding that xylene can selectively strip the PP layer. The stripping process is quantified by SEM-EDS data; sample data is shown in Table 3. This table compares SEM-EDS data taken from the inner surface of an EoL pouch, compared to an EoL pouch after 120 minutes of being soaked in xylene at 120 °C. While the inner surface before solvent treatment consists of 96% carbon (consistent with a PP layer), after solvent stripping the Al surface is clearly exposed.

As shown in Fig. 7(e), we also attempted to 'repair' some of the selectively stripped pouch material by covering it with a new PP film. A simple crosshatch adhesion test did indicate that there was some bonding between the aluminium surface and the PP film. To investigate this further, a new PP film was applied to (a) a pouch film where the PP layer had been fully stripped (using limonene at 90 °C for 60 min on an EoL pouch film) and (b) a pouch film where the PP layer had only been partially





Fig. 7 Photographs of (a) an edge piece of a pouch cell that has been opened, (b) an edge piece after soaking in xylene at 120 °C for 2 h, (c) the inner surface of a pristine pouch piece (approx 25 mm long) after 10 min in xylene at 90 °C, (d) the inner surface of a pristine pouch piece (approx 25 mm long) after 120 min in xylene at 120 °C, (e) the inner surface of a pouch after stripping in xylene and coating in a new PP layer, (f) a cross-hatch test on an Al-PP sample, and (g) PP that has been stripped from the inner pouch surface before being precipitated out by the addition of acetone. The left vial contains PP that had been recovered from a pristine pouch, while the right vial contains PP that had been recovered from an EoL pouch.

stripped (limonene at 90 °C for 10 min on an EoL pouch film). A cross section was cut through each of the 'repaired' materials and SEM images were taken of the two cross sections. Images of the cross sections are shown in Fig. 9. Neither sample showed perfect bonding between the Al film and the PP (and this is to be expected, since in the manufacturing process there is an adhesion layer containing acid-modified PP to bind the PP to the aluminium. For an industrial recycling process, this binding layer would require further optimisation). In each case, some cracking and voids between the Al layer and the polymer film were observed. Qualitatively, the amount of voids and cracks observed were fewer in the case of the PP attached to the material that had only partially been stripped of the original PP layer. This finding may have important implications for future recycling processes in this area, since the ability to not only selectively strip certain layers from the multi-layer structure, but to only *partially* strip certain layers, may actually make the repair/recycle procedure more effective.

3 Experimental

Xylene and Limonene were obtained from Sigma Aldrich and used as received, TMO was synthesised adapting a published route, carrying out reactive distillation at 112 °C using 1 kg of 2,5-dimethylhexane-2,5-diol (Sigma Aldrich) and 1 g of HCZB 30 (Clariant, calcined at 500 °C for 1 hour).³⁹

Pouch cells were opened manually under a fume hood, using a ceramic scalpel/scissors. Electrodes and separator materials were removed and separated using a bespoke suction device. All cell components (including the pouch material) were soaked, separately, in an IPA bath in ambient conditions



Fig. 8 SEM-EDS elemental maps of a cross-section through a pouch, for (a) a pristine pouch, and (b) a pristine pouch after treatment with xylene.



Table 3 SEM-EDS analysis of inner pouch surfaces. Figures given for each element are wt%

Element	Inner pouch surface	Inner pouch surface after stripping with xylene
C	96.4	19.57
O	3.35	1.67
Al	0.03	75.29
P	0.14	
Si	0.07	
F		2.61
Fe		0.73
Ca		0.05
Cr		0.08

for 24 h. Components were then dried in a vacuum oven at 50/75 °C at 100 mbar for 24 h.

DSC was carried out using a Mettler Toledo TGA/DSC 3+ STARE system, at a heating rate of 10 °C per min.

FT-IR spectra in Fig. 4 were obtained using a Bruker Tensor 27 IR and OPUS software. FT-IR spectra in Fig. 6 were obtained using a JASCO FT/IR 4200 system with ATR attachment.

SEM imaging was carried out using a Hitachi Tabletop Microscope TM3030Plus (Hitachi High-Tech Corporation, Japan). For analysis of the pouch materials, each sample was coated in AuPd, sputtered at 80 mA for 30 s under vacuum (0.01 mbar).

Thickness variation tests were carried out by placing some of each solvent in a vessel, which was then brought to the desired temperature. Several small (approx 2.5×2 cm) pieces of pouch material would then be placed in the vessel with the solvent. Before being placed in the vessel, a corner of each

piece was marked asymmetrically with a scalpel so that on removal from the solvent it would still be obvious which side was the inner pouch and which the outer, even after removal of a polymer layer. At various time intervals, a piece of pouch would be removed from the vessel, rinsed with acetone and dried, before its thickness was measured with a digital micrometer. For each combination of solvent/temperature, this process was repeated twice ('sample 1' and 'sample 2' on each graph).

For the 'repair' stage, pieces of pouch that had been stripped of PP were placed in contact with a film of pristine PP. This was then placed on a hotplate under a weight so as to keep conformal contact of the PP with the pouch. The hotplate was heated to 160 °C for 20 minutes, after which time the polymer had softened. The material was removed from the heat source and allowed to cool to room temperature before further tests were carried out.

4 Conclusions

This study demonstrates that it is possible to selectively remove some polymer layers from a multilayer material consisting of an aluminium sheet coated on one side in polypropylene and on the other side by a combination of polymers including PET. All three of the solvents tested (xylene, limonene and TMO) showed a propensity to strip the inner PP layer from the pouch while leaving the central aluminium foil and the outer polymer layer intact. Since both limonene and TMO carry fewer hazard warnings than does xylene, these data have implications for all future reprocessing procedures involving polypropylene, and so the usefulness of this study is not limited to the field of batteries. In particular, this study has implications for the recycling of multilayer structures, where it is economically more viable to repair part of the multilayer than to recycle them all.

It is interesting that the inner layer was more readily stripped from pouches that had previously been used in a battery than from the 'pristine' pouch film as received from the manufacturer; DSC and FT-IR analysis corroborates our assertion that this finding means that some parts of the polymer degrade during use. Our findings may also have further implications for 'repair' procedures using this material, since it may be more effective to bond a new PP layer to a partially stripped PP surface than to an aluminium surface.

Author contributions

Jean E. Marshall: conceptualization, methodology, data curation, writing – original draft. Bethany Middleton: methodology, investigation, writing – review and editing. Dominika Gastol: methodology, investigation. Roberto Sommerville: methodology, investigation. Con R. McElroy: investigation, methodology, resources. Emma Kendrick: funding acquisition, project administration, writing – review and editing. Vanessa Goodship: funding acquisition, project administration, writing – review and editing.

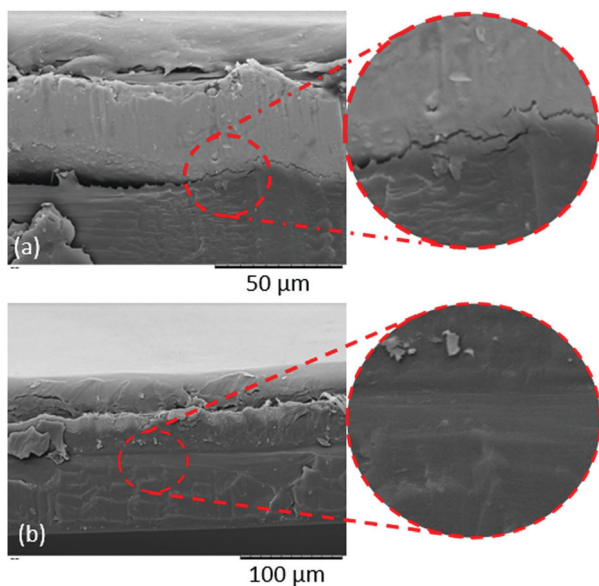


Fig. 9 SEM image of (a) a pouch film where the PP layer was fully stripped with limonene, before the application of a new layer of PP (b) a pouch film where the PP layer was only partially stripped with limonene before the application of the new PP layer. Insets show a zoomed-in part of the interface. Qualitatively, there appear to be fewer cracks and voids in image (b) than in image (a).



Conflicts of interest

There are no conflicts of interest to declare.

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