Institution: Loughborough University

Unit of Assessment: UOA8 - Chemistry

Title of case study: Closing the loop – Generating new plastic from old

Period when the underpinning research was undertaken: 2012-2020

Details of staff conducting the underpinning research from the submitting unit:

Name(s):	Role(s) (e.g. job title):	Period(s) employed by submitting HEI:
Prof. Steve Christie,	Professor of Chemical Technologies	1997-present
Prof. Paul Thomas,	Professor of Analytical Science	2007-present
Dr. Jim Reynolds,	Senior Lecturer	2007-present
Prof Colin Creaser,	Professor of Analytical Chemistry	2007-2017
Dr Matthew Turner	SEO/Lecturer	2013-present

Period when the claimed impact occurred: 2012-2020

Is this case study continued from a case study submitted in 2014? N

1. Summary of the impact (indicative maximum 100 words)

The environmental problems created by the production (from fossil fuels) and disposal of plastic are well known. Loughborough University's research, in collaboration with global leader in the plastics economy, *Plastic Energy (PE)*, identified a process through which useful products could be derived directly from end-of-life material. Loughborough refined the process to the point where it allowed the utilisation of a wider range of plastics and the controlled production of valuable, high purity materials for fuel and 'virgin' plastic production. This led to (i) commercial benefits for *PE* in the form of new contracts from international partners (£150M over 10 years) and (ii) environmental benefits via greater levels of recycling (tens of thousands of tonnes of plastic diverted from landfill).

2. Underpinning research (indicative maximum 500 words)

Plastics are ubiquitous to modern life. But their use comes at a price. Only 14% of plastic packaging is recycled, with the rest incinerated, land-filled, or ending up in the oceans (over eight million tonnes per annum). Loughborough University's research on volatile and combinatorial analysis of complicated mixtures has recently focused on the analysis of complex mixtures derived from crude oil and other related blends. The high accuracy and reproducibility of the range of advanced analytical techniques has resulted a robust suite of instruments capable of analysing complicated mixtures derived from fuel-type mixtures. It was this underpinning research that first attracted *Plastic Energy (PE)* to Loughborough in 2012. *PE*'s process addresses the chemical recycling of plastics via the pyrolysis of waste, end-of-life – and thus often contaminated - plastic for conversion via a thermal anaerobic process to produce naphtha and a diesel type fuel (*TACOIL*). This can be further converted to monomeric materials for recycling back to virgin plastics, hence "closing the loop". The process required detailed information on the composition of the various fractions, especially any potential contaminants that could be present.

By its nature, such work requires a multi-disciplinary approach – in this case, a synergy between organic and analytical chemistry. Prof Steve Christie's expertise in synthetic organic chemistry fed into all aspects of the production (and optimisation thereof) of organic materials generated within the study. Prof Paul Thomas and the team within the Centre for Analytical Science (Dr Jim Reynolds, Dr Matthew Turner and Prof Colin Creaser) have globally leading expertise that allowed characterisation of products at various stages of the process. A key indicator of this is shown in work that underpinned this project in which they demonstrated the ability to analyse the complex mixtures (thousands of compounds) present in crude oil via an

intricate combination of the FAIMS technique allied to fractionation, high resolution mass spectrometry and specific data analysis tools [R1] and [R2]. This was supported by work showing the capability to perform direct qualitative analysis, in real time, of oil systems via the DART-MS process the use of mass spectrometry to characterise crude oil components [R3]. Related techniques using UPLC-MS have been used in similarly complicated mixtures occurring in bio-diesel production [R4].

This precision analysis was used to support *PE* in efficient production of *TACOIL* and optimising its purity. Firstly, *PE*'s system required the close control of the pyrolysis process. Our investigation into the precise conditions required for optimisation meant that the chain lengths of the resulting molecules could be defined, maximising the yield of the *TACOIL* product. Secondly, general waste plastic from commercial and domestic sources contains a myriad of additives and other impurities. Part of the process of controlling such impurities was the development of methods for their accurate identification and quantification, and our work successfully addressed this issue. Working on a lab scale (100g) we carried out numerous experiments involving changing additives and refining the equipment setup, to optimise conditions. The team carried out detailed analysis of the end products to provide a robust picture of the diesel and *TACOIL* products, with results detailed to *PE* through confidential reports.

3. References to the research (indicative maximum of six references)

[R1] K.M. Szykuła, C. Wicking,S. Whitmarsh, C. S. Creaser and J. C. Reynolds "Characterization of crude oil and its saturate, aromatic and resin fractions by high-field asymmetric waveform ion mobility spectrometry high resolution mass spectrometry", *Energy Fuels* 2018, **32**, 11310–11316. doi.org/10.1021/acs.energyfuels.8b02718

[R2] C. Da Costa, M. Turner, J. C. Reynolds, S. Whitmarsh, T. Lynch, and C. S. Creaser "Direct Analysis of Oil Additives by High-Field Asymmetric Waveform Ion Mobility Spectrometry-Mass Spectrometry Combined with Electrospray Ionization and Desorption Electrospray Ionization"*, Analytical Chemistry*, 2016, **88**, 2453. doi.org/10.1021/acs.analchem.5b04595

[R3] C. Da Costa, S. Whitmarsh, T.Lynch and C.S.Creaser "The qualitative and quantitative analysis of lubricant oil additives by direct analysis in real time-mass spectrometry", *International Journal of Mass Spectrometry*, 2016, **405**, 24-31. doi.org/10.1016/j.ijms.2016.05.011

[R4] K. F. Haigh, G. T. Vladisavljevic, J. C. Reynolds, Z. Nagy, B. Saha "Kinetics of the pre-treatment of used cooking oil using Novozyme 435 for biodiesel production"*, Chemical Engineering Research and Design*, 2014, 713 doi.org/10.1016/j.cherd.2014.01.006

All papers were published in international journals following peer review. [R1] and [R2] were published ACS journals, with Analytical Chemistry being a flagship journal for this discipline. [R3] appeared in the International Journal of Mass Spectrometry, which is a key international resource for important result utilising this technique, while Chemical Engineering Research and Design [R4] is an official journal for the European Federation of Chemical Engineering and the Institution of Chemical Engineers.

4. Details of the impact (indicative maximum 750 words)

Loughborough research on the recycling of used plastic back to new – 'closing the loop' – was taken up by *Plastic Energy* (*PE*) a company the forefront of efforts to recycle old plastics directly into new ("Plastic2Plastic") and, working together, created a **knowledge transfer pathway to the current impacts.** To this end, *PE* decided to invest £300K to sponsor a PDRA

employed by Loughborough from 2012-2016 to initiate the process of building the equipment and ensure the relevant analytical procedures were robust and accurate. From late 2016, this PDRA transferred employment directly under *Plastic Energy*, but continued to physically work with the UoA at Loughborough.

Together, the team built a scale model of *PE*'s commercial plant was assembled, with appropriate analytical sampling points. The chemical process could then be followed in detail, allowing the manipulation of reaction conditions to achieve high efficiency and purity of product stream. This knowledge was transferred to the full-scale commercial plants, giving confidence that the new conditions could be transferred from the lab (100s of grammes) to the process scale (multiple tonnes). As noted in 2015 by David McNamara, Chief Technology Officer for *PE* **[S1]**:

> "We were keen to partner with Loughborough due to their expertise in both reactor design and specifically their knowledge in analysis of volatile organic mixtures. Our process produces a very complex mixture of volatile organic compounds, and detailed analysis of the product is vital to our success. Indeed, we were aware that the product stream would vary with subtle differences in the feedstock and reactor conditions, so we required a means to monitor this."

In a video release by the company he added **[S2]**:

> "Working with Loughborough has allowed us to look at the business on a molecular level and allows us to maximise the yield and quality if the fuels we produce from the plastics that are processed in our plants".

In the same release Andrew Lake, Principal Scientist at *PE*, confirmed **[S2]**:

> "…working to help optimise the process in particular what contaminants are present in the feedstocks, this is helping to redesign the plants in Europe".

To sustain their R&D efforts at Loughborough, *PE* further enhanced their operations by investing in two further two employees in 2019/2020. They currently operate two plants in Spain (with another under construction). In 2018 they signed up to "The New Plastics Economy Global Commitment", led by the *Ellen MacArthur Foundation*, with an undertaking to convert at least 300,000 tonnes of low-grade plastic waste into feedstock for new plastic manufacturing by 2025. In supporting *PE* to achieve this goal, we have **achieved the following impacts**:

Impact 1: Commercial benefit to *Plastic Energy* through new contracts with globally leading polymer producers

Loughborough University collaborated with *PE* to provide a robust and reliable method for the chemical recycling of end-of-life plastics. *PE* is a global leader in this area, specialising in the processing of waste plastic into a feedstock for generating clean recycled plastics or fuel. As *PE* note **[S1]**,

> "Loughborough has allowed the accelerated implementation of key developments, particularly the ability to identify key chemical trends in our process through advanced analytical techniques. This has given us the confidence to take developments at Loughborough from the small scale to our pilot plants which operate at the multi-tonne scale".

Two facets of making a process such as this commercially viable come in the form of (i) efficient production of the product and, concomitantly, (ii) optimisation of its purity. Both challenges have been met via our insights into *PE*'s technology. Firstly, the yield of the *TACOIL* product was maximised through Loughborough's analysis. Secondly, Loughborough's work produced a route that controls the production of deleterious by-products, especially chloride

and chlorine containing impurities. These particular impurities are notorious for their impact on the upcycling back into polymer, as there are threshold limits that must be met, otherwise the commercial value of the product plummets. This has allowed the formation of International Sustainability and Carbon Certification certified Circular Polymers in collaboration with SABIC and partners **[S3]**. This has 'closed the loop', upcycling mixed waste plastic back to original polymer.

After scaling Loughborough's experiments to the multi-tonnage level to ensure the same criteria have been met, *Plastic Energy* is now a reliable partner to major companies such as *SABIC* **[S4]**, *Sealed Air* **[S5]**, *TOTAL* **[S6]** and *Ineos* **[S7]**. These are globally-leading polymer and fuel manufacturers and/or users that produce millions of tonnes of material per annum, and *PE* supply them with *TACOIL* for up upcycling to polymers, and/or recycling of their polymer products. As *PE* note,

> "Loughborough has assisted with the formation of key industrial collaborations and partnerships including the development of new plastic waste processing facilities…." **[S1]**

Impact of the wider adoption of the *PE* process through major partnerships worldwide is evidenced in **[S4-7]**. To quote the company:

> "Loughborough has contributed to signing of global contracts with major companies in the area of plastic recycling. These are of a value over £150M and over 10 years, and have helped secure *Plastic Energy* as a leading proponent of plastic recycling worldwide." **[S1]**

The work has also impacted commercially locally, through *PE*'s opening of labs on Loughborough's LUSEP Enterprise Park in order to continue research and development.

Impact 2: Environmental benefit via a greater level of recycling

Our research underpinned the *Plastic Energy* process which has enabled the closed loop recycling of end-of-life plastics [R1-4]. This has clear environmental benefits since any plastic that is recycled does not go to landfill. These plastics have also re-entered the plastic cycle and take the place of new fossil fuel derived starting materials. This reduces the amount of fossil fuel required, reducing the carbon footprint of the entire plastics process. This is a major industrial and governmental policy in many countries, and one that the oil industry is being forced to adhere to.

Plastic Energy's process allowed the oil industry to comply with this. Plastic Energy estimated that they have diverted tens of thousands of tonnes from landfill over the period. This occurred at *PE*'s plants in Spain, with further impact in Europe via the adoption of the process in collaborations with *SABIC* (Netherlands site) **[S4]**, *Total* (France **[S6]** and *Ineos* (Germany) **[S7]**

PE noted the work thus far has impact:

> "…including the development of new plastic waste processing facilities and the diversion of tens of thousands of tonnes of waste plastic from landfill/incineration." **[S1]**

5. Sources to corroborate the impact (indicative maximum of 10 references)

[S1] Letter of support, from David McNamara, Chief Technology Officer, *PE*. (2020)
[S2] *PE* Partnership video transcript, showing collaboration and partnership in research and development (2015)
[S3] Press Release showing International Sustainability and Carbon Certification certified Circular Polymers in collaboration with SABIC and partners, (2019).

[S4] *PE/SABIC* Partnership Press Release confirms strategic relationship and plant facility in the Netherlands. (2020)
[S5] *PE/Sealed Air* Partnership Press Release on Strategic Collaboration (2020)
[S6] *PE/Total* Partnership Press Release: development of a new 15k tonnes p.a. plant in France (2020)
[S7] *PE/Ineos* Partnership Press Release confirms completion of advanced recycling process, and upscaling via new plant (2020)