Institution: University of Leeds

Unit of Assessment: UoA 8 Chemistry

Title of case study: Keracol Limited: a University of Leeds spin-out developing and commercialising sustainable personal care products

Period when the underpinning research was undertaken: 2002-date

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:
Professor Christopher Rayner	Professor of Organic Chemistry	1/10/1989-date

Period when the claimed impact occurred: 2014-date

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

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

Keracol is a University of Leeds spin-out that commercialises cosmetics and personal care products based on sustainable technologies. Research at the University of Leeds allows the extraction of high-value ingredients from food-waste through patented techniques, which are used in mass-production of skin and haircare products, through Keracol's own brand (Dr Craft) and with multinational partners (M&S Pure Super Grape), [text removed for publication] saving over 24.3 tonnes of fruit waste as of 2019. New patented hair dyeing technology invented by Keracol is being developed in partnership with global leaders [text removed for publication] to replace currently-used chemicals [text removed for publication].

2. Underpinning research (indicative maximum 500 words)

Professor Chris Rayner (Professor of Organic Chemistry, School of Chemistry) and Dr Richard Blackburn (Associate Professor in Sustainable Materials, School of Design; returned in UoA 32) have been collaborating on the development of sustainable cosmetic ingredients at the University of Leeds since 2002. Rayner's expertise in extraction techniques and processing has allowed the development of sustainable methods to yield the ingredients found in these materials in larger quantities than previously possible. Blackburn's background in sustainable dyes and historical dyeing techniques has brought new insights into the range of applications possible using natural materials. Specifically, they have developed expertise in the sustainable isolation, fractionation and identification of colourants from a range of natural sources, applying rigorous characterisation protocols to identify and quantify the exact components in the mixtures; for example, development of an improved extraction protocol for the European madder plant allowed the separation and first crystallographic characterisation of the anthraquinone colourant lucidin primoveroside. **[1]**

A key goal of their research has been to develop methods to valorise waste products, especially food waste. Investigations through projects at the University of Leeds funded by DEFRA (2007-9) and Innovate UK (then Technology Strategy Board; with Critical Processes Ltd., 2010) led to the development of technologies for the organic solvent-free extraction of natural antioxidants (such as resveratrol) and anthocyanin colourants from the skins of grapes and blackcurrants. The skins of these fruits constitute significant waste streams of wine and fruit juice/cordial production respectively and represent significant opportunities for valorisation. For example, an aqueous and solid-phase extraction was used to process waste blackcurrant pomace from the production of Ribena and the resulting components were fully characterised. **[2]** An integrated extraction/adsorption protocol was developed and found to deliver improved yields of anthocyanins of higher quality, while offering savings in time and energy: the results were found to be scalable for industrial production. **[3]** Quantitative studies on the adsorption behaviour of anthocyanin extracts from blackcurrant waste on human hair demonstrated the potential for application of the

extracts as commercial hair colourants. **[4]**

Blackburn and Rayner have continued to investigate other sources of sustainable raw materials for cosmetic applications. Examples include: how citrus waste from mandarin canning in China can be used as a key ingredient in new applications (through a BBSRC Newton Award with Keracol and partners); the use of novel extraction methods to isolate components from liquorice for applications in skincare, particularly natural UV absorbers (BBSRC, with Keracol and Neal's Yard Remedies; 2016-17); extraction of natural polyphenols, polysaccharides and colourants from seaweed (Innovate UK, with Bod Ayre, a seaweed farming company; 2009-11) **[P2]**; and the use of enzymes to esterify anthocyanins with fatty acids to allow their formulation for oil-based cosmetics and food applications. **[5]**

Work has also continued to seek and apply novel technologies to the problem of hair colouration. Exploiting their synergistic expertise in catalysis (Rayner) and colouration technology (Blackburn) (e.g. in integrated dye/catalyst systems for the production of coloured poly(lactic) acid **[6]**), new catalysed systems for the oxidative colouration of hair have been developed that enable the removal of the most hazardous synthetic dye ingredients and still allow a full shade range with permanent wash performance. Patents assigned to the University of Leeds have been granted extensively for this technology. **[P3]**

Intellectual property (including **[P1-3]**) developed through the above research led to the establishment of Keracol Limited in 2011 as a spin-out company from the University of Leeds.

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

The quality of the underpinning research is evidenced by (a) outputs [1]-[6] being published in international peer-reviewed journals and (b) the citations (Web of Science, 14/1/21) of each output that support their influence and scientific impact within the community.

[1] "Isolation and extraction of lucidin primeveroside from *Rubia tinctorum* L. and crystal structure elucidation" R. L. Henderson, C. M. Rayner, R. S. Blackburn, *Phytochemistry*, **2013**, *95*, 105-108. DOI: 10.1016/j.phytochem.2013.07.001. (9 citations)
Development of an improved extraction protocol allows separation and characterisation of natural dyes from the European madder plant.

[2] "Enhancing the Potential Exploitation of Food Waste: Extraction, Purification, and Characterization of Renewable Specialty Chemicals from Blackcurrants (*Ribes nigrum* L.)" S. Farooque, P. M. Rose, M. Benohoud, R. S. Blackburn, C. M. Rayner, *J. Agric. Food. Chem*., **2018**, *66*, 12265-122273. DOI: 10.1021/acs.jafc.8b04373. (4 citations)
Characterisation and quantitation of anthocyanins and other components from the organic-free extraction of blackcurrant waste from Ribena production.

[3] "Extraction of anthocyanins from *Aronia melanocarpa* skin waste as a sustainable source of natural colorants" M. H. Wathon, N. Beaumont, M. Benohoud, R. S. Blackburn, C. M. Rayner, *Coloration Technology*, **2019**, *135*, 5-16. DOI: 10.1111/cote.12385. (8 citations)
Joint publication between University of Leeds and Keracol Limited describing a novel and improved extraction/adsorption protocol for anthocyanin extraction.

[4] "Application of Anthocyanins from Blackcurrant (*Ribes nigrum* L.) Fruit Waste as Renewable Hair Dyes" P. M. Rose, V. Cantrill, M. Benohoud, A. Tidder, C. M. Rayner, R. S. Blackburn, *J. Agric. Food. Chem*., **2018**, *66*, 6790-6798. DOI: 10.1021/acs.jafc.8b01044. (16 citations)
Joint publication between University of Leeds and Keracol describing the extraction of anthocyanin colourants from waste blackcurrant skins and demonstration of applicability in hair dyeing.

[5] "Selective enzymatic lipophilization of anthocyanin glucosides from blackcurrant (*Ribes nigrum* L.) skin extract and characterization of esterified anthocyanins" L. Cruz, M. Benohoud, C. M. Rayner, N. Mateus, V. de Freitas, R. S. Blackburn, *Food Chemistry*, **2018**, *266*, 415-419. DOI:

10.1016/j.foodchem.2018.06.024. (15 citations)
Joint publication between University of Leeds and Keracol describing enzymatic methods to acylate natural anthocyanin colourants from blackcurrant skins.

[6] "The combined synthesis and coloration of poly(lactic) acid" R. O. MacRae, C. M. Pask, L. K. Burdsall, R. S. Blackburn, C. M. Rayner, P. C. McGowan, *Angew. Chem. Int. Ed.*, 2011, *50*, 291-294. DOI: 10.1002/anie.201004920. (17 citations)
Development of integrated catalysts/colorants for production of coloured plastics, which technology underpins the technologies claimed in patents [P3].

Granted patents:
[P1] R. S. Blackburn, P. M. Rose, C. M. Rayner, *Natural Hair Dyes*. Australian Patent 2010247136 (2/2/2017), Canadian Patent 2835316 (31/7/2018), European Patent 2477597 (3/10/2018), United States Patent 8361167 (29/1/2013).
[P2] R. S. Blackburn, C. M. Rayner, *Personal care composition comprising a natural film-forming biopolymer and methods of making the same.* Australian Patent 2013369087 (12/7/2018), European Patent 2934682 (17/10/2018).
[P3] R. S. Blackburn, C. M. Rayner, C. Pask, P. C. McGowan, *Catalysed Dye Systems*. Australian Patent 2010268006 (22/12/2016), Canadian Patent 2803942 (5/2/2019), Chinese patent 102470080 (29/8/2017), European Patent 2448545 (7/9/2016) and 3111915 (4/10/2017), South Africa Patent 201200796 (31/10/2012), United States Patent 8535391 (17/9/2013).

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

Keracol Limited was established as a spin-out of the University of Leeds in August 2011 to exploit the technologies developed by Rayner and Blackburn for the development of sustainable and safe personal care product ingredients. Impact has been demonstrated in the review period (2014-date) through: continued growth and development of the business; introduction of new products to the market (by Keracol and in partnership with a multinational business); positive impact on production through reducing waste / improving resource efficiency; and change of product design within a multinational company.

New products derived from food waste
University of Leeds research has delivered new processes for extracting cosmetic ingredients and colourants from sustainable sources, especially from food waste. **[2, 3, P1, P2]** Keracol have taken products based upon these sustainably sourced ingredients to market, both on their own and in partnership. They have commercialised technology from Leeds for the extraction from grape skins of resveratrol, a natural anti-oxidant frequently used in skin care products for its anti-ageing properties. Keracol partnered with the multi-national Marks and Spencer to create a new range of products containing resveratrol extracted from its own English grape waste (Pinot Noir produced for M&S by Chapel Down Vineyard in Kent) using the Leeds technology. In 2014 M&S launched a new range of skincare products using this sustainably-sourced resveratrol under the name Pure Super Grape and continues to sell these through M&S stores and online. **[A]** [text removed for publication] The Pure Super Grape range has had a positive impact on production through reduced waste, with Keracol confirming it "*has saved 17.3 tonnes of grape pomace (waste) from entering UK landfill*" between 2014-2019. **[B, C]**
In 2018 Keracol launched their own brand, Dr Craft **[D]** (as a joint venture with Ellipsis Limited, who lead on sales, marketing and distribution), exploiting Leeds research on the extraction of colourants from blackcurrant skins (a major waste product in the production of fruit juices and cordials) **[2, 4, P1]** and citrus waste. The product range includes shampoo, conditioner and a hair brightening serum, which are sold online through Amazon. The product range attracted significant media attention **[E]** and won top prize for New Sustainable Product at the 2019 Sustainable Beauty

Awards. **[F]** The positive impact on reducing waste is confirmed by Keracol's technical director who states that "*production of extracts for our products has saved 5 tonnes of blackcurrant waste and 2 tonnes of mandarin waste from landfill or incineration*" in the two years of sales to date. **[C]**

Change of product design within a multinational company

Keracol has also developed technology to significantly improve the safety of synthetic hair dyes. Many current hair dyes involve the use of *para*-phenylenediamine, which is known to be a contact allergen and has raised issues as a potential carcinogen. Keracol licenses patented technology developed at the University of Leeds by Rayner and Blackburn **[P3]** for a novel catalyst that enables the removal of the most hazardous synthetic hair dye ingredients, but still enables a full shade range with permanent wash performance. This technology is currently being commercialised in partnership with [text removed for publication] one of the largest personal care companies in the world [text removed for publication]. **[C]**

In the review period, Keracol has demonstrated impact through launched commercial products and reduced waste. Their commercial portfolio continues to grow (face creams and oils in the Dr Craft range based on bakuchiol as the anti-oxidant ingredient have already been launched **[D]**) and the ongoing design and delivery of new products is supported by demonstrable collaborations between the University of Leeds, Keracol and other industrial partners (e.g. a hairspray using components extracted from seaweed **[P2]**, developed through a collaborative TSB grant with BodAyre).

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

[A] https://www.cosmeticsdesign-europe.com/Article/2014/07/24/Marks-Spencer-skin-care-range-from-grape-waste-product
[B] Annual sales figures of Pure Super Grape products from Technical Director, Keracol, Leeds, UK (28th September 2020)
[C] Letter of corroboration from Technical Director, Keracol, Leeds, UK (28th September 2020)
[D] https://drcraft.co.uk
[E] See, for example, https://www.bbc.co.uk/news/uk-england-leeds-44303626
[F] https://www.cosmeticsdesign.com/Article/2019/11/26/And-the-winners-of-the-2019-Sustainable-Beauty-Awards-are
[G] [text removed for publication]
[H] [text removed for publication]