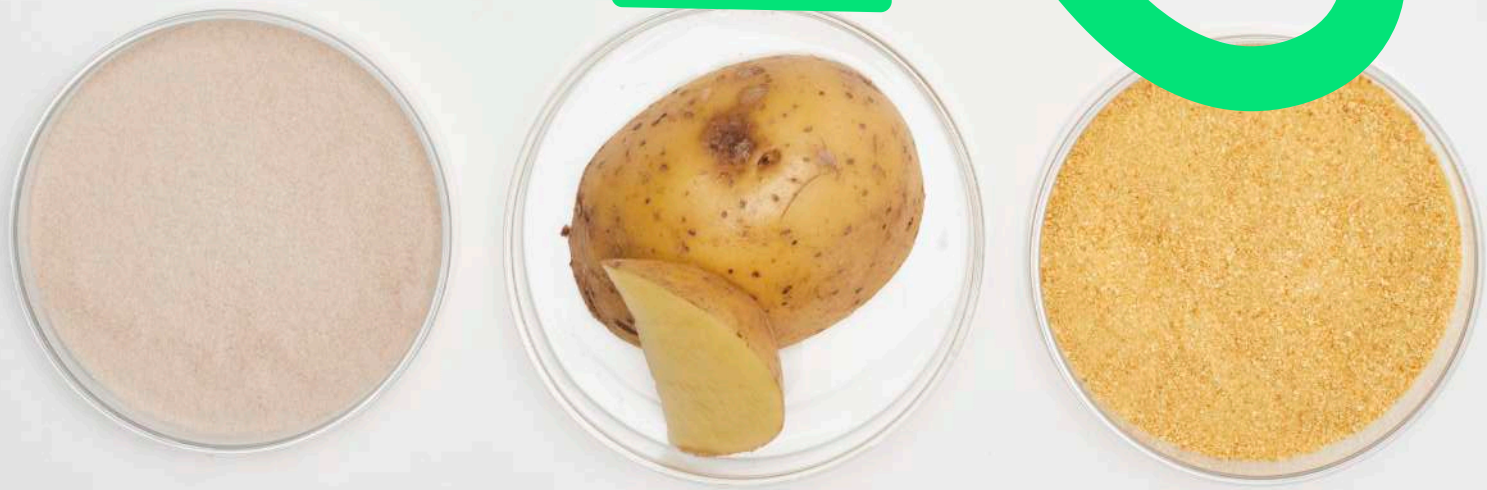
Reflow

constRuctive mEtabolic processes For materiaL fIOWs in urban and peri-urban environments across Europe

Material Development & Properties Report

REFLOW
MATERIOM



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REFLOW MATERIOM

Material Development & Properties Report

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TABLE OF CONTENTS

Reflow

I. INTRODUCTION

A. Introduction

REFLOW is an EU Horizon 2020 research project running from 2019 to 2022, aiming to enable European cities' transition towards circular and regenerative practices. REFLOW uses Fab Labs and maker spaces as catalysers of a systemic change in urban and peri-urban environments.

The project has provided best practices aligning market and government needs to create favourable conditions for the public and private sectors to adopt circular economy (CE) practices. REFLOW is creating new CE business models within six pilot cities: Amsterdam, Berlin, Cluj-Napoca, Milan, Paris and Vejle, and assess their social, environmental and economic impact by enabling active citizen involvement and systemic change to re-think the current approach to material flows in cities.

II. KEY BIOPOLYMERS

A. City Unavoidable Food Waste

On the basis of the research conducted by Metabolic and Materiom for the different REFLOW pilot cities, large volumes of unavoidable food waste (UFW) were identified in all project cities. In particular, the research focused on Milan, Vejle and Amsterdam because of their potential alignment with biomaterials.

The model developed by Metabolic determined the avoidable and unavoidable volumes of food waste based on FAO data (2011, 2017) and relevant literature (De Laurentiis et al., 2018; John-Jaja et al., 2016; WRAP, 2014). Materiom then analysed the UFW volumes and determined the potential availability of biopolymers in the waste that could be relevant for biomaterial production. In the following diagram, one can review the number of tons of unavoidable food waste for Milan.

Table 1 - Unavoidable Food Waste Milan Diagram - Metabolic

B. Biopolymer Availability

The number of biopolymers available in the UFW can be reviewed in this second diagram. The analysis conducted by Materiom identified the potential raw amount available for each type of biomass. However, the amount of biopolymer eventually extracted may vary depending on the extraction method used and the variety of fruit, vegetables or animals.

Table 2 - MILAN Biopolymer Availability per UFW tons Diagram - Materiom

Unavoidable Waste	*Tons*	*Biopolymer*	*Tons*
Coffee & Products	5349.46	Cellulose	460.05
		Hemicellulose	1,963.25
Poultry Meat	4971.03	Collagen (Gelatine)	745.65
Pigmeat	2617.80	Collagen (Gelatine)	157.07
Oranges, Mandarines	2472.34	Cellulose	283.08
		Hemicellulose	269.49
		Pectin	443.79
Bananas & Plantains	1974.38	Cellulose	236.93
		Starch	296.16
		Pectin	493.59
Eggs	1415.23	Calcium Carbonate	1,344.46
Potatoes & Products	1030.86	Starch	206.17
Bovine Meat	769.40	Collagen (Gelatine)	46.16
Lemons, Limes & Products	631.49	Pectin	99.14
Onions	423.15	Cellulose	190.42
		Hemicellulose	84.63
Apples and products	419.94	Cellulose	37.00
		Hemicellulose	22.84
Pineapples and products	376.84	Cellulose	63.91
		Hemicellulose	46.13
		Pectin	11.53
Grapes and products (excl wine)	163.67	Cellulose	17.19
		Hemicellulose	9.98
Tea (including mate)	135.20	Cellulose	21.63
Mutton & Goat Meat	86.64	Collagen (Gelatine)	745.65
Grapefruit and products	51.01	Pectin	10.97
Fish	14.58	Collagen (Gelatine)	1.46
Dates	10.22	Cellulose	8.59
		Pectin	0.61
Molluscs & Crustaceans	9.18	Calcium Carbonate	2.30
		Chitin	0.46

Source: Arbia et al., 2012; Homester et al., 2012; Hue, Minh Hang & Razumovskaya, 2017; Nys et al., 2004; Suresh et al., 2016; Szymańska-Chargot, 2017; Torres et al., 2020; Pareek, 2016; Prasad & Rhim, 2018; Rodriguez & Castro, 2019; Wongsiridetchai et al., 2018; Yang & Shu, 2014; Zhao et al., 2018.

From the analysis above, four biopolymers were chosen on the basis of the following criteria. First, **cellulose, gelatine and starch** were chosen because of the large amount of biopolymers available in the observed waste. This makes sense in order to valorise these resources that are considered waste, and to re-integrate them into a new life cycle. In addition, **chitin** was chosen because of the high market value of this biopolymer. While not available in large quantities at the urban UFW level, this biopolymer is the second most naturally abundant biopolymer in our biosphere, and there is a diverse literature demonstrating the desirable properties it can have when used for biomaterials. Moreover, it has been demonstrated as possible to harvest chitin from black soldier flies fed on mixed organic waste (Sanandiya et al., 2020), offering the potential to link its production to UFW.

B. Biopolymers

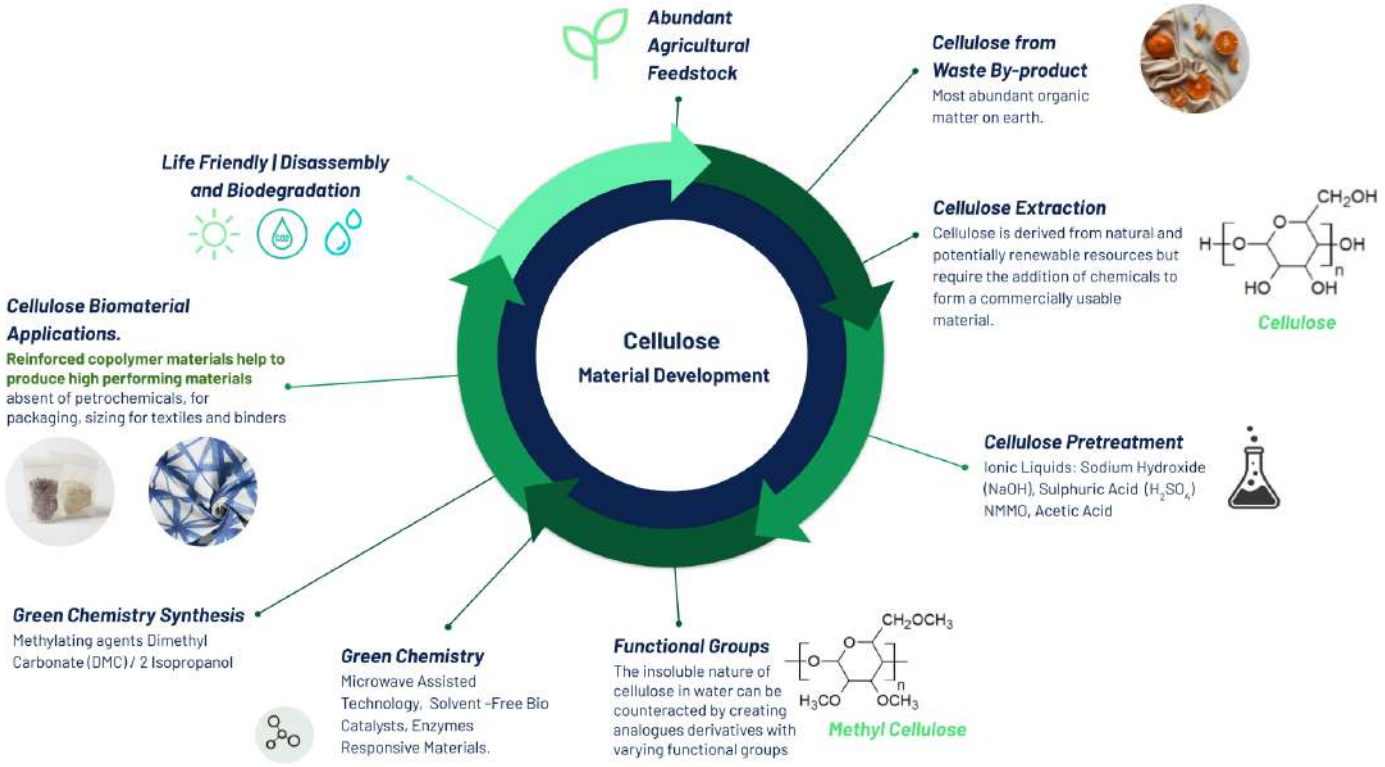
Cellulose

Cellulose is the most abundant naturally occurring polymer in the biosphere, which plants and many bacteria synthesise (Kaplan, 1998). Cellulose is formed by microfibrils with a stiff, ordered structure responsible for strength and resistance degradation, making it not soluble in water (Szymańska-Chargot, 2017).

Role in Nature: Structural reinforcement and strength

Role in Biomaterials: Filler, Reinforcing Material

Table 3 - Cellulose Material Development Diagram - Materiom



Gelatine

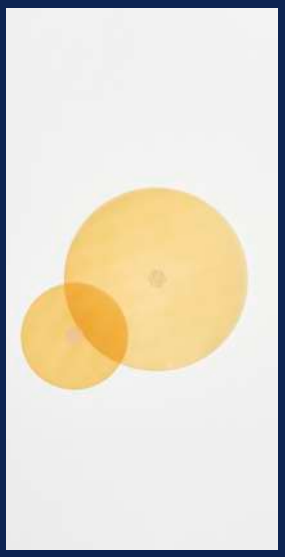
Gelatine comes from Collagen, a major structural protein in animal tissue and the most abundant protein in the animal kingdom.
Gelatine is a hydrocolloid, a substance that produces gel on contact with water, which in the case of gelatine is reversible (Schrieber, 2007).

Role in Nature: Intracellular matrix of connective tissues.
Role in Biomaterials: Structural Matrix, Binder, Hydrocolloid, Foaming agent.

Starch

Starch is a polysaccharide produced by most green plants for energy storage. Starch is of interest as a biopolymer material because of its low cost, its availability as an agricultural byproduct, and its thermal processability using conventional plastics processing equipment (Kaplan, 1998).

Role in Nature: Energy
Role in Biomaterials: Structural Matrix, Binder.



Chitin

Chitin and chitosan, a derivative of chitin, are very interesting polymers due to their mechanical properties and low general solubility (Rinaudo, 2006), critical attributes for films and materials (Kaplan, 1998).

Role in Nature: Structural reinforcement and strength
Role in Biomaterials: Structural Matrix, Binder.

III. MATERIAL DEVELOPMENT

A. Biomaterials systems

All biomaterial prototypes formulated for the pilot cities were produced adhering to Life Friendly Chemistry Principles (Dorfman, 2012). The biomaterial systems generated for the pilot cities were based on the material waste flow analysis discussed. These systems are as follows:

Table 4 - Biomaterial systems composition

1.1	*Methyl-cellulose* *Chitin*	–	*Glycerol*	*Water* *Vinegar*
2.8	*Methyl-cellulose* *Gelatin*	–	*Glycerol*	*Water*
3.1	*Methyl-cellulose* *Potato Starch*	–	*Glycerol*	*Water* *Vinegar*

Structural Matrix

Biopolymer that agglomerates the material

Filler

Fibre or mineral that fills in the material, enhancing specific properties

Plasticizer

Biopolymer that enhances the flexibility and strength of the material

Solvent

Dissolves the main components that promote chemical catalysis

These material systems were developed through co-polymerisation of the structural matrix precursor to provide tunable structural and mechanical properties based on variations in the component ingredients. Optimised formulations of the material systems in Table 3 were conducted utilising the following process techniques:

1.1 Chitosan and Methyl-cellulose Bioplastic

Tools

- Spatula
- Pot
- Hob
- Scale
- Thermometer
- Measuring jug
- Measuring spoons
- Moulds
- Dehydrator

Ingredients

Chitosan
10 g

Methyl-cellulose
10 g

Glycerol
7 g/ml

Water
275 g/ml

Vinegar
150 g/ml

Steps

Add the vinegar (150 g/ml) to distilled water (275g/ml) and glycerol (7g/ml), then stir until boiling.

Add chitosan (10 g) to the mixture stepwise in small portions, whilst stirring until dissolved into solution. Reduce the heat of the solution whilst adding chitosan to a simmer (65-75 ºC), and stir until homogenous.

Dissolve the Chitosan, add the methyl-cellulose (10 g) stepwise and stir in solution. You may still find small lumps over time depending on the grade of chitosan - Remove these with a spatula.

Pour into moulds, once all the solids have dissolved. Then leave to set in the air - once moderately set transfer to the dehydrator at 35 ºC for 18-21hrs.

2.8 Gelatin and Methyl-cellulose Bioplastic

Tools

- Spatula
- Pot
- Hob
- Scale
- Thermometer
- Measuring jug
- Measuring spoons
- Moulds
- Dehydrator

Ingredients

Gelatin
12 g

Methyl-cellulose
15 g

Glycerol
7 g/ml

Water
300 g/ml

Steps

Add distilled water (300 g/ml) and glycerol (7 g/ml) to the reaction vessel (beaker, or heat resistance pot), then stir until boiling.

Add methyl-cellulose (15 g) to the mixture stepwise (in small portions) whilst stirring until dissolved into solution, then proceed to add the gelatine (12 g) and stir into the solution

Reduce the heat of solution to simmering point (65-75 ºC) and stir until homogenous. Remove any lumps or froth with a spatula.

Pour into moulds, once all the solids have dissolved. Then leave to set in the air - once moderately set transfer to the dehydrator at 35 ºC for 18-21hrs.

3.1 Potato Starch and Methyl-cellulose Bioplastic

Tools

- Spatula
- Pot
- Hob
- Scale
- Thermometer
- Measuring jug
- Measuring spoons
- Moulds
- Dehydrator

Ingredients

Potato Starch
12 g

Methylcellulose
15 g

Glycerol
7 g/ml

Water
450 g/ml

Vinegar
20 g/ml

Steps

Add vinegar (20 g/ml) to distilled water (300 g/ml) and glycerol (7 g/ml), then stir until boiling.

Add starch to a small jar (10g) and stirred into cold water (150g/ml). The cold starch solution is added to the mixture once brought to a boil and stirred once the solution has dissolved. Then, add methyl-cellulose (15g) to the mixture stepwise (in small portions) whilst stirring until dissolved into the solution.

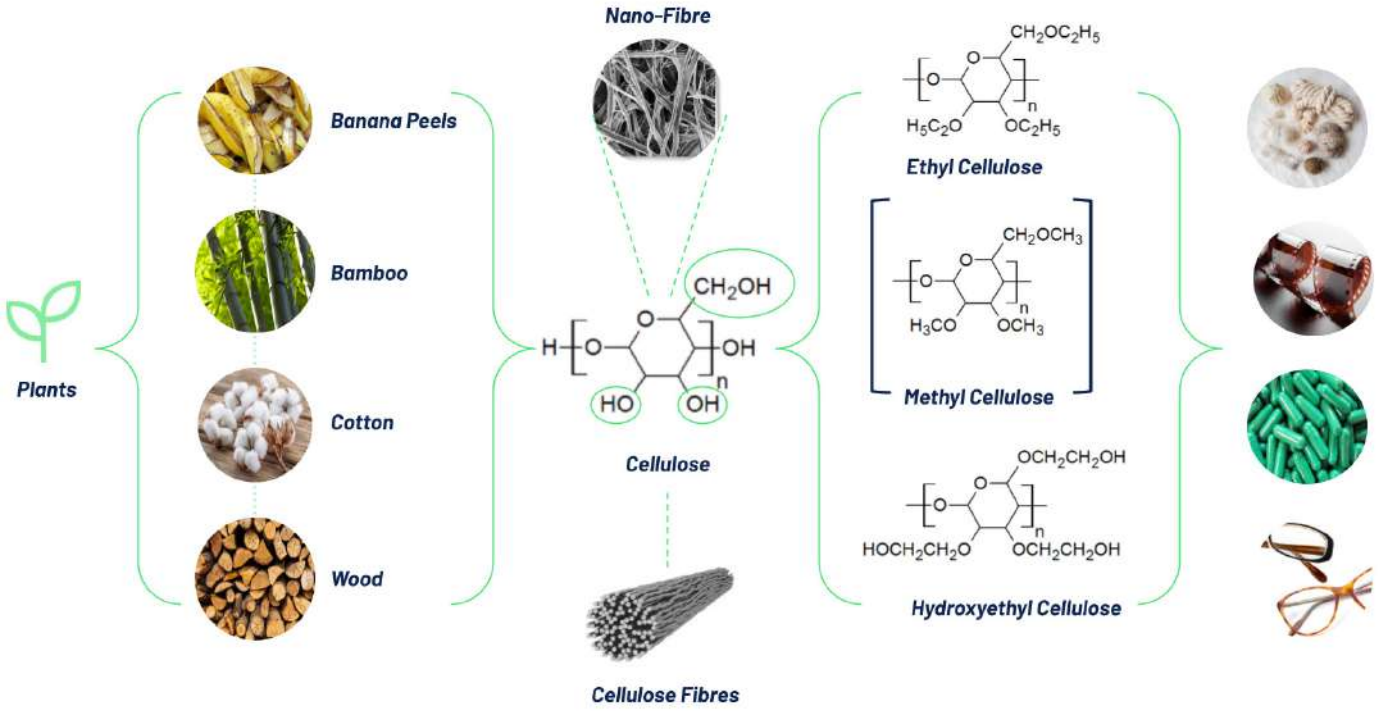
Reduce heat of the solution to a simmer (65-75 ºC) and stir until homogeneous. You may still find small lumps over time depending on the type of starch - Remove these with a spatula.

Pour into moulds, once all the solids have dissolved. Then leave to set in the air - once moderately set transfer to the dehydrator at 35 ºC for 18-21hrs.

B. Cellulose to Methyl-cellulose

Due to the **water soluble nature** of methyl-cellulose, it has been utilised in these studies as an accessible route to material making to demonstrate the versatility of cellulose and its derivatives in **forming biofilms with a broad range of properties for packaging and textile applications**. Furthermore, methyl-cellulose is currently exploited in the textile industry for sizing applications, protecting fibres from water and oil (Tan et al. 2019).

Table 5 - Cellulose R&D - Materiom



IV. PROPERTIES ANALYSIS & POTENTIAL APPLICATIONS

A. Properties analysis

The mechanical properties of each biomaterial prototype were assessed using a commercial **tensile testing machine.** Each material formulation is represented by a unique identification number to differentiate between variations in ingredient concentration within a material system, defined by a common set of ingredients and process steps. The Design of Experiment (DoE) approach to material synthesis facilitates the tuning of material properties required for a desired final application. The initial mechanical properties achieved show promise for application in packaging and textiles.

The data also indicates that with **increased concentration of each ingredient component, there is an increase in performance in the strength of the samples**. The strength and toughness of a material is defined by the following key factors: **strength** (hardness) signifies a material's resistance to irreversible deformation, (which is certainly true for ductile materials). However, **toughness** is defined by a material's resistance to fracture and is therefore assessed as the energy required to cause fracture to a material (Ritchie, 2011).

Notably, samples **Methyl-cellulose-Gelatin 2.8** and **Methylcellulose-Starch 3.1** show significant strength and toughness, complying with previously reported analogous systems for cellulose and and polysaccharide or amino acid polymerisations (Marichelvam et al., 2019; Yaradoddi, 2020), with **2.8 the strongest and 3.1 the toughest.** This may be attributed to the specific types of **biopolymers interacting within the structural matrix**, depending on the threshold of polysaccharides derived or protein derived material within the system. The **degree of plasticiser** also influences the interaction between the bonds thus the degree of flexibility which in turn influences the mechanical performance. Thus, by employing a Design of Experiments (DoE) model to the material development of each sample system we are able to derive an optimum performing biomaterial (see Appendix 1). Table 6, indicates the degree at which concentration variations within each component of the material system impacts the mechanical properties for each sample.

Table 6 - Biomaterial Design of Experiment

Co-biopolymers	Unique ID	Elongation at yield (%)	Max force (N)	Ultimate tensile stress (MPa)	Elongation at break (%)	Young's modulus (MPa)
Chitosan & M-Cellulose	1.1	2.39%	72.8	30.42	33.8%	489
	1.6	-	44.3	8.26	12.2%	-
	1.8	2.66%	74.4	15.75	24.3%	247
Gelatin & M-Cellulose	2.1	3.33%	76.6	32.11	14.8%	944
	2.6	2.71%	114.8	26.29	54.4%	372
	2.8	2.88%	133.5	33.82	9.6%	1092
Starch & M-Cellulose	3.1	2.27%	62.2	37.54	41.6%	772
	3.6	1.99%	88.0	24.65	42.3%	522
	3.8	2.21%	67.3	21.02	40.1%	453

Table 7 - Materials Ultimate tensile stress (MPa) Comparison

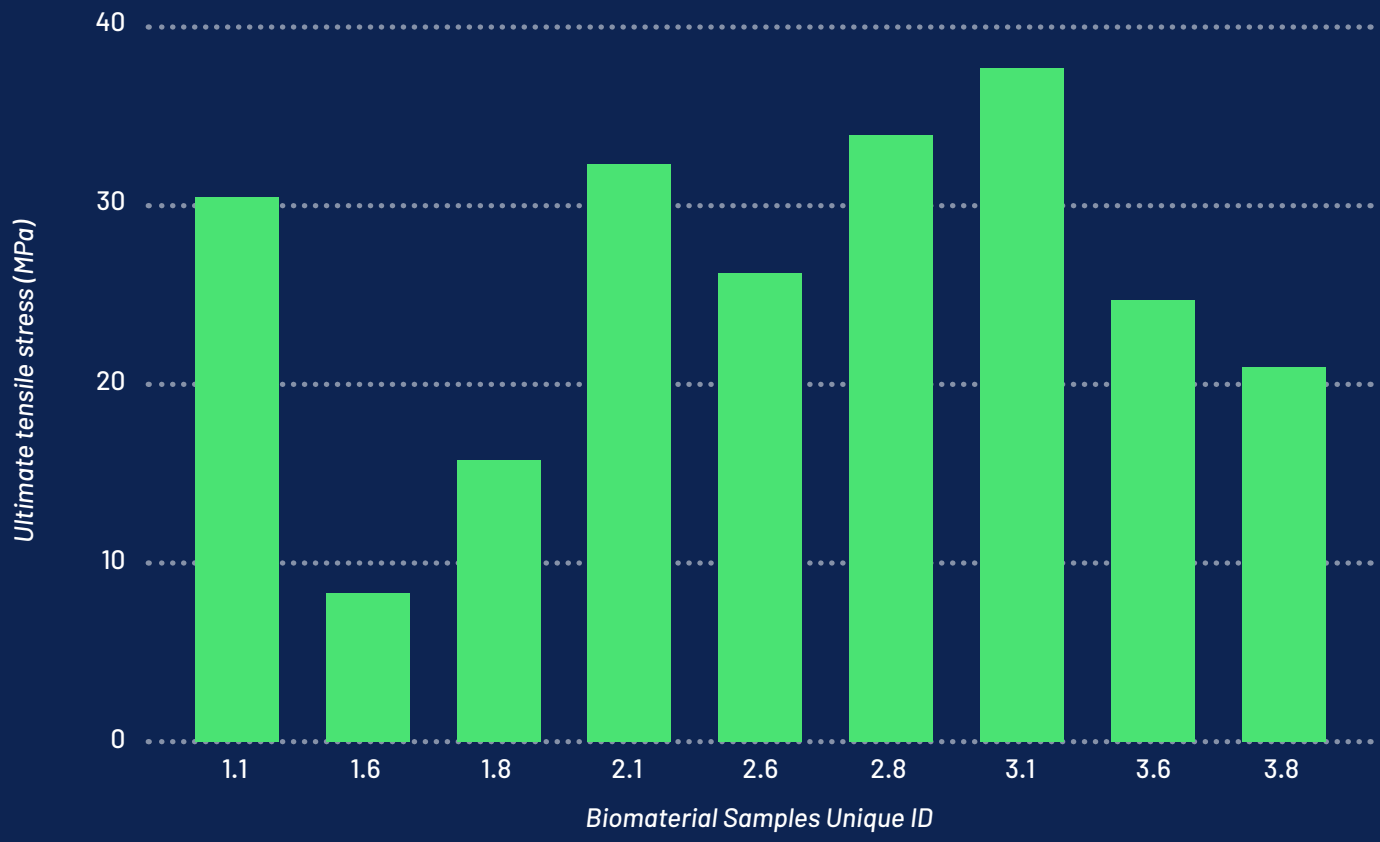
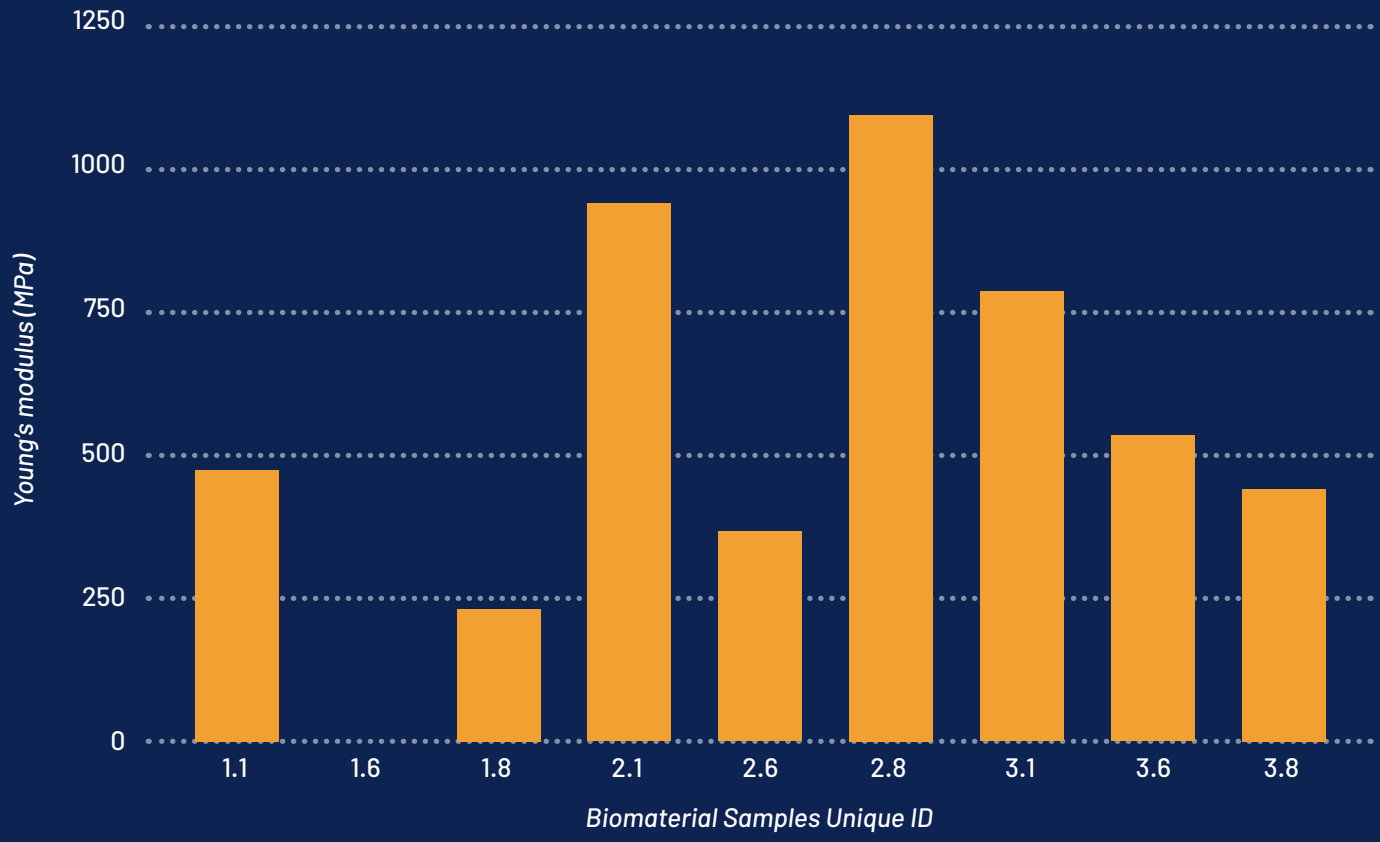


Table 8 - Materials Ultimate tensile stress (MPa) Comparison



B. Potential applications

Initial mapping of the mechanical properties for the material systems outlined in Table 3 can be used as a preliminary indication of their suitability for an intended application. Represented here (Figure 9) is an Ashby diagram which highlights the Young Modulus against strength (i.e. resistance to deformation or compression after an applied force maximum versus maximum strength a material can withstand upon elongation before breaking). The Ashby diagram maps the material properties of diverse material systems (Ashby, 2010). The results of the preliminary mechanical tests conducted on the material samples can utilise these plots to compare performance.

1 Megapascal (MPa) = 0.001 Gigapascal (GPa)

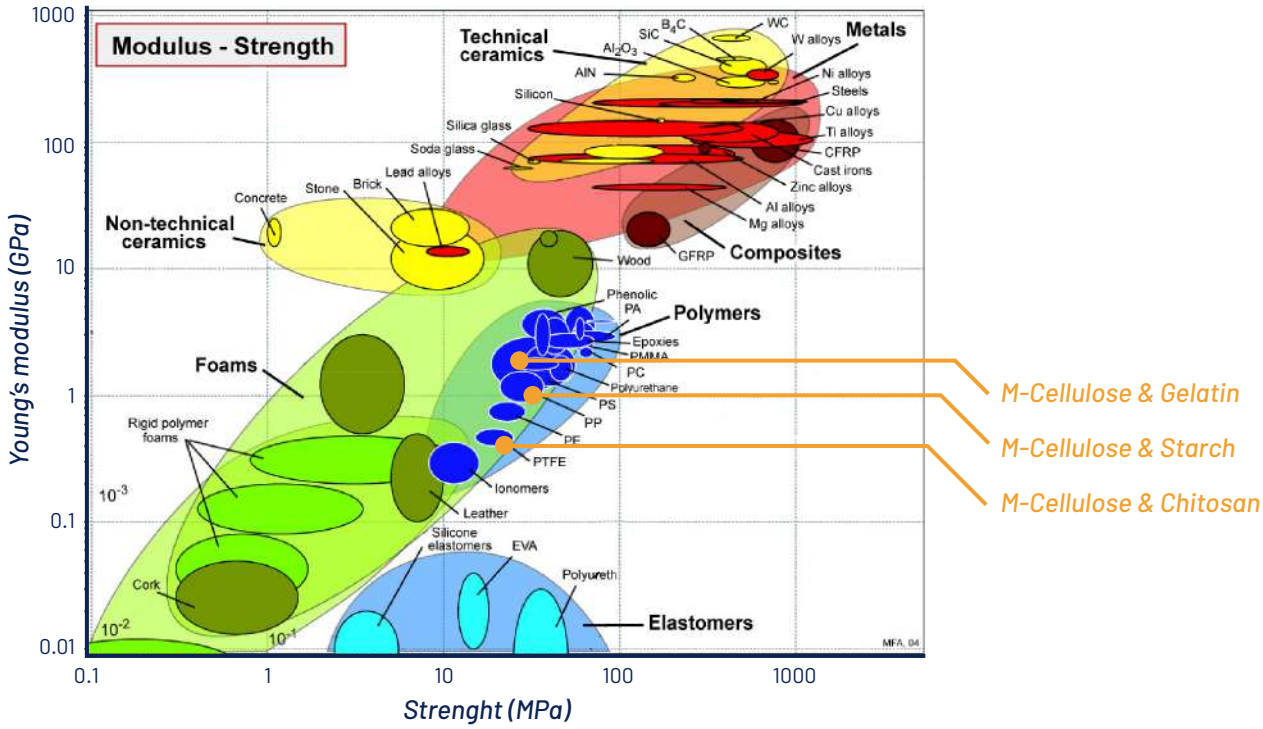
Highest Performing Samples:

1.1 Methyl-Cellulose Chitosan	*489MPa = 0.489 GPa*	*Comparative to Foam - Polymers area*
2.8 Methyl-Cellulose Gelatine	*1092MPa = 1.092 GPa*	*Comparative to Polymers area*
3.1 Methyl-Cellulose Starch	*772MPa = 0.772GPa*	*Comparative to Polymers area*

As depicted in figure 9 the prototyped material systems are comparative to the performance of known petrochemical derived plastic such as Polypropylene (PP), Polytetrafluoroethylene(PTFE), PMMA Poly(methyl methacrylate), utilised for packaging and textile applications (Sangroniz et al., 2019).

Figure 9 - Young's modulus against Strength.

Ashby (2010). Material and process charts. Chart 3.



** Regional Reference Point*

While the identification of applications requires further exploitation, the mechanical properties obtained suggest good alignment with packaging, interior upholstery, binders industries, and sizing for textiles, which is an intermediate technical process by which the yarn, fabric or textile is protected by a resin or adhesive (Wenqiang et al., 2019). This preliminary data show that the pairing of cellulose-derived compounds with an accompanying biopolymer provides an accessible route to developing high-performing reinforced materials absent of petrochemicals.

Further studies and material development can be implemented to investigate these materials' chemical and physical properties in more depth. These studies could allow for the expansion of non-toxic and high viscosity biomaterials for packaging applications that utilise green chemistry additives, alongside providing biodegradable protective or encapsulating coatings for textile fibres.

C. Other relevant industrial applications by biopolymer

The following applications are already found in commercial use (Gorgieva & Kokol, 2011; Kaplan, 1998; Rinaudo, 2006; Schrieber, 2007; Szymańska-Chargot, 2017). These usages could provide references for the further applications of the developed material systems.

Cellulose

- Polymers Development: films, adhesives and binders.
- Packaging Industry
- Textiles Fibres
- Manufacturing Resources: Sprayable and Moldable Material, Building insulation.

Collagen/ Gelatin

- Biotechnology: Tools and Products development
- Consumer Products, such as Packaging or Glue
- Textiles: Coating, Protection, and Products

Starch

- Consumer Products, such as Glues and Packaging (as a film or cushioning foam)
- Polymers Development, such as PLA
- Textiles: Maintenance of clothes, reduce yarn breaking during weaving, textiles printer thickener
- Biofuel

Chitin

- Biopolymers development
- Feedstock for manufacturing processes

V. DIGITAL FABRICATION PROCESSING

Based on the characteristics of the developed biomaterial systems, the following digital fabrication processes are recommended to develop possible applications in the design and creative industries. *For more recipes and techniques please review www.materiom.org*

Laser cutter

Vector Cutting

Lugae

For modules and joints

Half-Vector Cutting

Lugae

For structuring and work with volumens

Vector Cutting & Engraving

Emilce Cesarini

For flexibility and textures

Laser Scanning

Margarita Talep

Textured Moulds

Clara Davis

3D printer

Solution Printing

Mould Printing

Composite Printing

If filler is added

Router CNC

Mould Making

VI. CONCLUSION

This report aims to analyse and illustrate the possibility of approaching material production from a circular regenerative approach within the Reflow project. Biomaterials are highlighted as an opportunity to use untapped resources from cities' waste flows, specifically unavoidable food waste streams.

The biomaterial systems developed based on four notable biopolymers could be an appropriate starting point for designers, makers and SMEs that want to start working in this area. With the information gathered in this report, creatives and entrepreneurs can make these materials, and explore the properties and characteristics that can be obtained compared to commercial materials in the market.

There are many remaining challenges in creating circular models around these materials. It is necessary to analyse the whole supply chain, the energy consumption, and the implications of scaling up production. Moreover, from a life-friendly chemistry perspective, it is deemed necessary to study further the Life Cycle Assessment of material biodegradation, disassembly via microorganisms, enzyme technology or biochemical breakdown, subjected to surface and degradation experiments, and durability additives.

Our aim is to provide designers, makers and entrepreneurs with a solid starting point for biomaterial innovation that helps enable a circular and regenerative economy.

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VIII. APPENDIX

APPENDIX 1 - Material Experiments

Each material system was subjected to a Design of Experiment methodology for optimised results.

Table 10 - Chitosan & M-Cellulose Experiments

Sample	Run	Starting Material (g)		Plasticiser (g/ml)	Solvent (g/ml)	Acid (g/ml)
	Name	M-Cellulose	Chitosan	Glycerol	Water	Acetic Acid/ Vinegar
1.1	-1	10 (-1)	10 (-1)	7 (-1)	400	30
1.2	a	20 (+1)	10(-1)	7(-1)	400	30
1.3	b	10(-1)	20(+1)	7(-1)	400	30
1.4	ab	10 (-1)	10(-1)	15(+1)	400	30
1.5	c	20(+1)	20(+1)	7(-1)	400	30
1.6	ac	20(+1)	10(-1)	15 (+1)	400	30
1.7	bc	10(-1)	20(+1)	7(-1)	400	30
1.8	abc	20(+1)	20(+1)	15(+1)	400	30

Table 11 - Gelatin & M-Cellulose Experiments

Sample	Starting Material (g)		Plasticiser (g/ml)	Solvent (g/ml)
	M-Cellulose	Gelatin	Glycerol	Water
2.1	15(-1)	12 (-1)	7 (-1)	250
2.2	20 (+1)	12(-1)	7(-1)	250
2.3	15(-1)	24(+1)	7(-1)	250
2.4	15 (-1)	12(-1)	15(+1)	250
2.5	20(+1)	24(+1)	7(-1)	250
2.6	20(+1)	12(-1)	15 (+1)	250
2.7	15(-1)	24(+1)	7(-1)	250
2.8	20(+1)	24(+1)	15(+1)	250

Table 12 - Starch & M-Cellulose Experiments

Sample	Starting Material (g)		Plasticiser (g/ml)	Solvent (g/ml)	Acid
	M-Cellulose	Starch	Glycerol	Water	Vinegar
3.1	15 (-1)	5 (-1)	7 (-1)	650	10
3.2	25 (+1)	5(-1)	7(-1)	650	10
3.3	15(-1)	10(+1)	7(-1)	650	10
3.4	15 (-1)	5(-1)	15(+1)	650	10
3.5	25(+1)	10(+1)	7(-1)	650	10
3.6	25(+1)	5(-1)	15 (+1)	650	10
3.7	15(-1)	10(+1)	7(-1)	650	10
3.8	25(+1)	10(+1)	15(+1)	650	10

APPENDIX 2 - Material Properties

Each sample was run twice to get a mean average.

Table 13 - Physical measurement for each sample (Thickness, sample width and cross section)

Unique ID	Sample ID	Sample thickness (mm) (random locations)					Sample width (mm)			Cross sectional area (m^2)
		Meas. 1	Meas. 2	Meas. 3	Meas. 4	Avg.		Meas. 2	Avg.	
1.1	1.1A	0.23	0.24	0.26		0.24	8.5	8.3	8.40	2.04E-06
1.1	1.1B	0.24	0.29	0.30		0.28	8.8	8.5	8.65	2.39E-06
1.6	1.6A	0.54	0.57	0.54	0.60	0.56	8.0	8.0	8.00	4.50E-06
1.6	1.6B	0.56	0.69	0.62		0.62	8.5	8.7	8.60	5.36E-06
1.8	1.8A	0.57	0.65	0.60	0.57	0.60	8.0	7.8	7.90	4.72E-06
1.8	1.8B	0.66	0.68	0.67		0.67	8.2	8.5	8.35	5.59E-06
2.1	2.1A	0.26	0.29	0.27	0.33	0.29	8.2	8.4	8.30	2.39E-06
2.1	2.1B	0.38	0.36	0.23		0.32	8.3	8.1	8.20	2.65E-06
2.6	2.6A	0.62	0.69	0.59	0.59	0.62	8.0	8.2	8.10	5.04E-06
2.6	2.6B	0.53	0.55	0.47		0.52	8.7	8.2	8.45	4.37E-06
2.8	2.8A	0.54	0.52	0.45	0.40	0.48	8.5	8.5	8.50	4.06E-06
2.8	2.8B	0.52	0.48	0.41		0.47	8.5	8.3	8.40	3.95E-06
3.1	3.1A	0.22	0.20	0.18	0.18	0.20	8.5	8.5	8.50	1.66E-06
3.1	3.1B	0.20	0.26	0.28		0.25	8.2	8.0	8.10	2.00E-06
3.6	3.6A	0.48	0.43	0.40	0.40	0.43	8.2	8.5	8.35	3.57E-06
3.6	3.6B	0.49	0.38	0.46		0.44	8.5	8.8	8.65	3.83E-06
3.8	3.8A	0.41	0.31	0.34	0.36	0.36	8.7	8.7	8.70	3.09E-06
3.8	3.8B	0.35	0.36	0.42		0.38	8.5	8.5	8.50	3.20E-06

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