D4.9. Demonstration products produced delivered to industrial end users for market test

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Contents

Executive summary

This deliverable summarizes the final products produced and delivered to industrial end users for market test based on feed stocks of rapeseeds, tomatoes, and citrus fruits.

1. INTRODUCTION

During the Pro-Enrich project, different biorefinery processes have been developed from feedstocks of olives, rapeseeds, tomatoes and citrus fruits. Some of these processes have led to several Pro-Enrich products that are being tested in formulated market products by end users. The objective of this deliverable is to provide an overview of products produced and delivered to industrial end users for market test based on feedstocks of rapeseeds, tomatoes and citrus fruits. Some of the products described here were previously reported in D4.2. and have been updated in this deliverable. The following table features a summary of all the samples produced by each partner involved. The testing of samples is reported in D5.6 *Final performance assessment report all end users* due in M35.

Sample name	Raw material	Partner	Amount
Carotenoids-rich tomato oleoresin	Cherry tomatoes	Natac	20 g
Carotenoids-rich tomato powder	Cherry tomatoes	Natac	50 g
Protein hydrolysate from tomato seeds	Tomato pomace	BU	40 g
Citrus, dry extract, 60% Hesperidin	Orange peel	Natac	300 g
Citrus, dry extract, 90% Hesperidin	Orange peel	Natac	300 g
Protein extract from hot-pressed rapeseed cake (batch 1)	Rapeseed	DTI	5 kg
Protein extract from hot-pressed rapeseed cake (batch 2)	Rapeseed	DTI	5 kg
Protein extract from cold-pressed rapeseed cake (batch 4)	Rapeseed	DTI	0.1 kg
Protein extract from cold-pressed rapeseed cake (batch 5)	Rapeseed	DTI	0.5 kg
Protein extract from cold-pressed rapeseed cake (batch 8)	Rapeseed	DTI	1 kg
Protein extract from cold-pressed rapeseed cake (batch 11)	Rapeseed	DTI	1 kg
Protein extract from cold-pressed rapeseed cake (batch 16)	Rapeseed	DTI	0.7 kg
Protein extract from hot-pressed rapeseed cake (batch 17)	Rapeseed	DTI	5.5 kg

2. PRODUCTS OBTAINED FROM TOMATOES

PRODUCT SPECIFICATIONS

Product: CAROTENOIDS-RICH TOMATO OLEORESIN
Product Code: NTPRL01
Botanical name: *Solanum lycopersicum*
Plant Part Used: Fruit (Pomace)
Description: Dark-red oily extract
Observations: Extraction of cherry tomato pomace with 2-MeTHF, filtration, concentration and vacuum drying

ANALYSIS	SPECIFICATION	METHODS
Assay (%)	Min 5,0 Lycopene	HPLC
Assay (%)	Min 7,0 Total carotenoids	Internal method
Loss on drying (%)	≤ 5,0	Eu. Pharm c.v. (2.8.17)
Microbiology		
TAMC (cfu/g)	≤ 10000	Eu. Pharm c.v. (2.6.12)
TYMC (cfu/g)	≤ 100	Eu. Pharm c.v. (2.6.12)
Bile-tolerant gram-negative bacteria (cfu/g)	≤ 100	Eu. Pharm c.v. (2.6.31)
Escherichia coli (1 g)	Absence	Eu. Pharm c.v. (2.6.31)
Salmonella (25 g)	Absence	Eu. Pharm c.v. (2.6.31)
Polycyclic aromatic hydrocarbons (PAHs) *		
Benzo(a)pyrene (ppb)	≤10,0	GC-MS
PAH4 (Sum of (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene (ppb)	≤ 50,0	GC-MS
Heavy metals*		
Lead (ppm)	≤ 3,0	Eu. Pharm. v.v. (2.4.27)
Arsenic (ppm)	≤ 2,0	Eu. Pharm. v.v. (2.4.27)
Mercury (ppm)	≤ 0,1	Eu. Pharm. v.v. (2.4.27)
Cadmium (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Pesticides*	According to Regulation (EC) Nº 396/2005 and amendments	SANTE/12682/2019

Storage: Store at 4ºC in the dark
Country of origin: Spain
Observations: Unlikely to be hazardous although tests have not been performed; unlikely to be hazardous to the environment

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The information set out in this specification and/or in any other document provided with the products does not constitute any warranty other than conformity to the current product specifications. The authorized uses of our products are not the same in all countries and it is your responsibility to verify that the use/s for which they are intended and their labelling are in accordance with current local or national legislation and regulations. The buyer agrees to hold the seller harmless against any third-party claim that it brings cause of unauthorized use of the product and/or its incorrect labelling.

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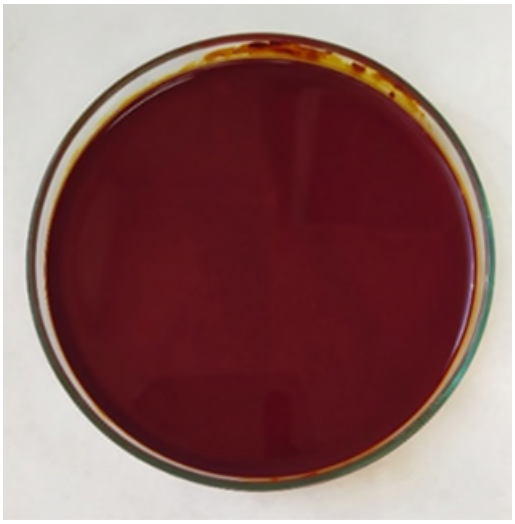

Figure 1. Carotenoids-rich tomato oleoresin.

PRODUCT SPECIFICATIONS

Product: CAROTENOIDS-RICH TOMATO POWDER
Product Code: NTPRL02
Botanical name: *Solanum lycopersicum*
Plant Part Used: Fruit (Pomace)
Description: Dark-orange powder
Observations: Extraction of cherry tomato pomace with 2-MeTHF, filtration, concentration and vacuum drying. Addition of drying agent, milled and sieved

ANALYSIS	SPECIFICATION	METHODS
Assay (%)	Min 1,0 Lycopene	HPLC
Assay (%)	Min 1,5 Total carotenoids	Internal method
Loss on drying (%)	≤ 5,0	Eu. Pharm c.v. (2.8.17)
Bulk density (g/ml)	≥ 0,3	Eu. Pharm c.v. (2.9.34)
Particle size (%)	Min 90% < 250 µm	Eu. Pharm c.v. (2.9.12)
Microbiology		
TAMC (cfu/g)	≤ 10000	Eu. Pharm c.v. (2.6.12)
TYMC (cfu/g)	≤ 100	Eu. Pharm c.v. (2.6.12)
Bile-tolerant gram-negative bacteria (cfu/g)	≤ 100	Eu. Pharm c.v. (2.6.31)
Escherichia coli (1 g)	Absence	Eu. Pharm c.v. (2.6.31)
Salmonella (25 g)	Absence	Eu. Pharm c.v. (2.6.31)
Polycyclic aromatic hydrocarbons (PAHs) *		
Benzo(a)pyrene (ppb)	≤10,0	GC-MS
PAH4 (Sum of (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene (ppb)	≤ 50,0	GC-MS
Heavy metals*		
Lead (ppm)	≤ 3,0	Eu. Pharm. v.v. (2.4.27)
Arsenic (ppm)	≤ 2,0	Eu. Pharm. v.v. (2.4.27)
Mercury (ppm)	≤ 0,1	Eu. Pharm. v.v. (2.4.27)
Cadmium (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Pesticides*	According to Regulation (EC) Nº 396/2005 and amendments	SANTE/12682/2019

Storage: Store at 4ºC in the dark
Country of origin: Spain
Observations: Unlikely to be hazardous although tests have not been performed; unlikely to be hazardous to the environment

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The information set out in this specification and/or in any other document provided with the products does not constitute any warranty other than conformity to the current product specifications. The authorized uses of our products are not the

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Figure 2. Carotenoids-rich tomato powder.

Technical Data Sheet			
1. Sample ref.	BU34.21	**2. Date of sampling**	29.6.2021
3. Sample name	Protein hydrolysate from tomato seeds		
4. Plant origin	Tomato pomace after removing juice (50 kg) - supplied frozen from Agrofusion		
5. Country of origin	Ukraine		
6. Processing	Sedimentation of seeds and skimming off peel and pulp on surface. High shear mixing of seeds and hydrolysis with Neutrase at neutral pH, centrifugation and rotary evaporation.		
7. Appearance	Pale yellow powder	**5. Quantity**	*40 g*
8. Ingredients	Protein content 35.9%		
9. Hazards	Unlikely to be hazardous although tests have not been performed; unlikely to be hazardous to the environment		
10. Storage	Keep dry at room temperature		
11. Comments	Crude protein hydrolysate from tomato seeds for testing by end-users of the consortium. 		

3. PRODUCTS OBTAINED FROM CITRUS

Natac Innovaoleo

PRODUCT SPECIFICATIONS

Product: **CITRUS, DRY EXTRACT, 60% HESPERIDIN**
Product Code: N202114901
Botanical name: *Citrus sp.*
Plant Part Used: Fruit (Peel)
Description: Yellowish-brown powder with characteristic odor and taste
Observations: Extraction of citrus peels with water (pH 11.5, Ca(OH)2). Clarification and acidification to pH 4.5 to precipitate the hesperidin. The extract was dried, milled and sieved.

ANALYSIS	SPECIFICATION	METHODS
Assay (%)	Min. 60,0 Hesperidin	HPLC
Loss on drying (%)	≤ 5,0	Eu. Pharm c.v. (2.8.17)
Bulk density (g/ml)	≥ 0,3	Eu. Pharm c.v. (2.9.34)
Particle size (%)	Min 95% < 250 microns	Eu. Pharm c.v. (2.9.12)
Residual solvents		
Ethanol (ppm)	< 5000	Eu. Pharm. v.v. (2.4.24)
Microbiology		
TAMC (cfu/g)	≤ 10000	Eu. Pharm. v.v. (2.6.12)
TYMC (cfu/g)	≤ 100	Eu. Pharm. v.v. (2.6.12)
Bile-tolerant gram-negative bacteria (cfu/g)	≤ 100	Eu. Pharm. v.v. (2.6.31)
Escherichia coli (1 g)	Absence	Eu. Pharm. v.v. (2.6.31)
Salmonella (25 g)	Absence	Eu. Pharm. v.v. (2.6.31)
Polycyclic aromatic hydrocarbons (PAHs) *		
Benzo(a)pyrene (ppb)	≤10	GC - MS
PAH4 (Sum of (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene (ppb)	≤ 50	GC-MS
Heavy metals*		
Lead (ppm)	≤ 3,0	Eu. Pharm. v.v. (2.4.27)
Arsenic (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Mercury (ppm)	≤ 0,1	Eu. Pharm. v.v. (2.4.27)
Cadmium (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Pesticides*	According to Regulation (EC) Nº 396/2005 and amendments	SANTE/12682/2019

Storage: Keep dry at room temperature
Country of origin: Spain
Observations: The product is not considered a hazardous material accord-ing to GHS (Global Harmonized System) and doesn't fall under Regulation (EC) 1272/2008 and CFR 1910 1200

Specification version: 000

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Figure 3. Citrus, dry extract, 60% Hesperidin**.**

PRODUCT SPECIFICATIONS

Product: **CITRUS, DRY EXTRACT, 90% HESPERIDIN**
Product Code: N21008403
Botanical name: *Citrus sp.*
Plant Part Used: Fruit (Peel)
Description: Yellowish-brown powder with characteristic odor and taste
Observations: Extraction of citrus peels with water (pH 11.5, Ca(OH)2). Clarification and acidification to pH 4.5 to precipitate the hesperidin. Dissolution of the precipitate in water (pH 12.0) and recrystallization (pH 4.7). Drying, milling and sieving.

ANALYSIS	SPECIFICATION	METHODS
Assay (%)	Min. 90,0 Hesperidin	HPLC
Loss on drying (%)	≤ 5,0	Eu. Pharm c.v. (2.8.17)
Bulk density (g/ml)	≥ 0,3	Eu. Pharm c.v. (2.9.34)
Particle size (%)	Min 95% < 250 microns	Eu. Pharm c.v. (2.9.12)
Residual solvents		
Ethanol (ppm)	< 5000	Eu. Pharm. v.v. (2.4.24)
Microbiology		
TAMC (cfu/g)	≤ 10000	Eu. Pharm. v.v. (2.6.12)
TYMC (cfu/g)	≤ 100	Eu. Pharm. v.v. (2.6.12)
Bile-tolerant gram-negative bacteria (cfu/g)	≤ 100	Eu. Pharm. v.v. (2.6.31)
Escherichia coli (1 g)	Absence	Eu. Pharm. v.v. (2.6.31)
Salmonella (25 g)	Absence	Eu. Pharm. v.v. (2.6.31)
Polycyclic aromatic hydrocarbons (PAHs) *		
Benzo(a)pyrene (ppb)	≤10	GC - MS
PAH4 (Sum of (benzo(a)pyrene, benz(a)anthracene, benzo(b)fluoranthene and chrysene (ppb)	≤ 50	GC-MS
Heavy metals*		
Lead (ppm)	≤ 3,0	Eu. Pharm. v.v. (2.4.27)
Arsenic (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Mercury (ppm)	≤ 0,1	Eu. Pharm. v.v. (2.4.27)
Cadmium (ppm)	≤ 1,0	Eu. Pharm. v.v. (2.4.27)
Pesticides*	According to Regulation (EC) Nº 396/2005 and amendments	SANTE/12682/2019

Storage: Keep dry at room temperature
Country of origin: Spain
Observations: The product is not considered a hazardous material accord-ing to GHS (Global Harmonized System) and doesn't fall under Regulation (EC) 1272/2008 and CFR 1910 1200

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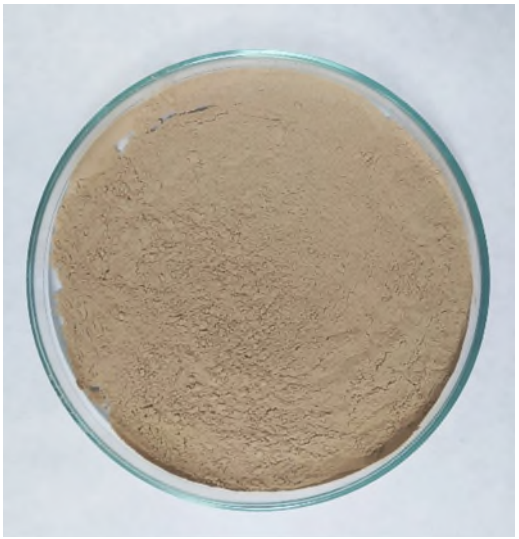
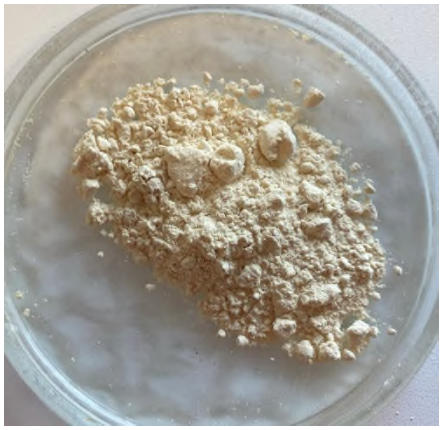


Figure 4. Citrus, dry extract, 90% Hesperidin**.**

4. PRODUCTS OBTAINED FROM RAPE SEEDS

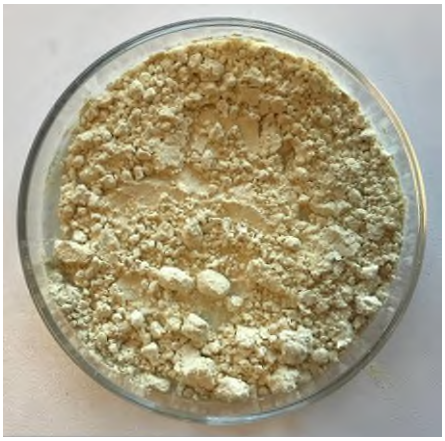
Technical Data Sheet (updated from D4.2)			
1. Sample ref.	Pro-Enrich; demo batch no. 01; sample no. 4-1	**2. Date of sampling**	02/2019
3. Sample name	Protein extract from hot-pressed rapeseed cake		
4. Appearance	Light yellow very fine powder Pleasant smell	**5. Quantity**	5 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibers, organic acids, phenols, lipids Protein: 29.5 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein extract from hot pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet (updated from D4.2)			
1. Sample ref.	Pro-Enrich; demo batch no. 02; sample no. 4-2	**2. Date of sampling**	02/2019
3. Sample name	Protein extract from hot-pressed rapeseed cake		
4. Appearance	Light yellow very fine powderPleasant smell	**5. Quantity**	5 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids Protein: 26.7 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein extract from hot pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no. 04; sample no. 4-1	**2. Date of sampling**	04/2019
3. Sample name	Protein extract from cold-pressed rapeseed cake		
4. Appearance	Dark yellow very fine powderPleasant smell	**5. Quantity**	0.1 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids Protein: data not available		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein extract from hot pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no.05; sample no. 4-1	**2. Date of sampling**	06/2019
3. Sample name	Protein concentrate from cold-pressed rapeseed cake		
4. Appearance	Dark yellow very fine powder Pleasant smell	**5. Quantity**	0.5 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids, tannin Protein: 56.6 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein isolate from cold pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no. 08; sample no. S011	**2. Date of sampling**	05/2020
3. Sample name	Protein concentrate from cold-pressed rapeseed cake		
4. Appearance	Light yellow very fine powder	**5. Quantity**	1 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids, tannin Protein: 70.8 % Lipid: 0.3 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein concentrate from cold pressed rapeseed cake for demonstration and testing by end-users of the consortium.		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no. 11; sample no. S010	**2. Date of sampling**	03/2020
3. Sample name	Protein isolate from cold-pressed rapeseed cake		
4. Appearance	Light yellow very fine powder	**5. Quantity**	1 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids, tannin Protein: 91.8 % Lipids: <0.3 % Sugars: 3 - 4% Ash: 3 - 4 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein isolate from cold-pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no. 16; sample no. S011	**2. Date of sampling**	07/2021
3. Sample name	Protein extract from cold-pressed rapeseed cake		
4. Appearance	Light yellow very fine powder Sticky during handling Pleasant smell	**5. Quantity**	0.7 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids, tannin Protein: 30 % (preliminary results)		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein extract from cold-pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

Technical Data Sheet			
1. Sample ref.	Pro-Enrich; demo batch no. 17; sample no. S016	**2. Date of sampling**	07/2021
3. Sample name	Protein concentrate from hot-pressed rapeseed cake		
4. Appearance	Dark yellow very fine powder Pleasant smell	**5. Quantity**	5.5 kg
6. Ingredients	Lignocellulose: Protein, sugars, dietary fibres, organic acids, phenols, lipids Protein: 72.8 %		
7. Hazards	Inhalation may cause allergic reaction; unhazardous to the environment		
8. Storage	Keep dry at room temperature		
9. Comments	Protein concentrate from hot pressed rapeseed cake for demonstration and testing by end-users of the consortium. 		

5. CONCLUSIONS

Several products have been successfully extracted by partners Danish Technological Institute, Natac and Bangor University. The functionality of the products is being tested by end users for specific applications: pet food, food, adhesives and/or cosmetics.