

Potato peel valorization through ethanolamine reactive organosolv treatment

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Abstract: Approximately 30% of the global potato supply is processed, generating substantial amounts of potato peel (PP) as a byproduct. Potato peel is a suberoligneous biomass, commonly found in the barks and peels of tuberous roots and tubers, and remains underutilized despite being a valuable source of cutin and suberin. The primary challenge in valorizing these compounds lies in the complexity of their extraction. To address this, an organosolv pretreatment based on ethanolamine aminolysis of esters was investigated. The ethanolamides produced from suberin deconstruction were thoroughly characterized using Fourier transform infrared (FTIR) spectroscopy, nuclear magnetic resonance (NMR) spectroscopy, and gas chromatography–mass spectrometry (GC–MS), and the results indicated that agricultural byproducts such as potato peels have the potential to diversify the sources of biobased compounds for diols in polymers, foam boosters, and bioactive materials, offering a sustainable alternative feedstock.

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Key words: potato peel; valorization; suberin; biobased; organosolv; ethanolamine; ethanolamides

Introduction

Globally, potatoes are consumed either raw or processed into ready meals, chips, and fries. Approximately 30% of potatoes undergo processing, generating 10% to 12% of their weight as a byproduct known as potato peels (PP).¹ The PPs contain carbohydrates such as starch, hemicellulose, and cellulose; other important components are lignin and lipids.¹

The valorization of wastes from the agrofood industry is a popular topic in both academic and industrial research.² The

biorefinery of lignocellulosic biomass has advanced through various processing routes. However, the use of suberoligneous biomass from the barks and peels of tuberous roots remains limited, with research primarily focusing on extracting lignin, starch, secondary metabolites, and energy production.³ Potato peels are valuable sources of polymers like cutin and suberin but they are not well utilized.⁴

Suberin has the potential to produce several million tons of biopolyesters rich in long-chain polyfunctional fatty acids.⁵ Given the stagnant production of fatty acids and the projected 6% compound annual growth in demand,⁶ extracting suberin

from agricultural byproducts such as peels could provide a sustainable alternative source. However, fully harnessing suberin requires the development of efficient extraction methods and the expansion of its existing applications.¹

A number of methods, such as transesterification, have been reported for the extraction of suberin;⁷ however, none of them satisfies all the requirements for practical industrial application.⁸ Transesterification works well but using a catalyst makes the process more expensive and complex and produces more effluents during the extraction process. For this reason, a new method was investigated, influenced by the chemical recycling of oil and polyethylene terephthalate.^{9,10} In particular, the ethanolamine (EA) aminolysis of ester serves as the basis for the process.¹¹ Ethanolamine has recently attracted interest in lignocellulosic biorefineries due to its unique chemical properties.^{12,13} A dual-functional solvent can act as a hydrogen bond donor and acceptor as well as a proton/hydrogen ion acceptor in a range of chemical reactions. In the biorefinery, this special mixture of features offers an extremely powerful potential. Suberin deconstruction by aminolysis produces ethanolamides,^{14,15} which can be used as biobased, biodegradable emulsifiers and surfactants with negligible adverse effects.¹⁶

Ethanolamides have gained increased attention, particularly from the food, pharmaceutical, and cosmetics industries,¹⁷ as environmentally friendly alternatives to common petroleum-based compounds.¹⁸ Some of them are also considered endogenous lipid mediators – biological mediators with roles in the cardiovascular, neurological, and immunological systems.¹⁹

Materials and methods

Materials

All of the experiments were based on potatoes that were available on a local market (Carrefour, Milan, Italy). Ethanolamine was purchased from Merck (Darmstadt, Germany), glycerol, ethyl acetate and sodium sulfate from

Sigma-Aldrich (St. Louis, MO, USA), ethanol from VWR International (Radnor, PA, USA).

Methods

Potato peels were treated in water (10% w v⁻¹) at 100 °C for 2 h in order to remove the residual starch. The suspension was filtered using a Büchner funnel, and the solid was lyophilized and milled. Approximately 500 mg of potato peel powder was treated with 5 mL of a glycerol–ethanolamine mixture at varying concentrations (Table 1) for 2 h at 120 °C and 10% w v⁻¹ consistency under magnetic stirring. The suspension was then filtered through a Büchner funnel and washed with 10 mL each of ethanol and water. The resulting solid was dried, weighed, and designated as a ‘cellulose-rich’ material. The filtrate was rotavaporized to remove the ethanol, acidified with 2 mol L⁻¹ HCl to pH 1, and extracted three times with 25 mL of ethyl acetate. After anhydrication with Na₂SO₄, filtration, and rotary evaporator, the yellow oil material was weighed and named ‘extractives’.

Fourier transform infrared spectroscopy (FTIR) was performed with a Nicolet iS10 (Thermo Fisher Scientific, Waltham, MA, USA) equipped with an attenuated total reflectance (ATR) sampling accessory with a diamond crystal (Smart iTR, Thermo Fisher Scientific). For each spectrum, 32 scans with a spectral resolution of 2 cm⁻¹ were recorded. Proton nuclear magnetic resonance (¹H-NMR) spectra were recorded using a Bruker Avance (Bruker, Billerica, MA, USA) 400 MHz solution NMR spectrometer. Around 10 mg of each sample was dissolved in 750 µL of CDCl₃ (Merck) and placed in a 5 mm NMR tube. The ¹H NMR spectra were acquired with the following parameters: 90° pulse width 10 ms, spectral width 12 ppm and relaxation delay 2 s. The total number of scans for every experiment was 64 (four dummy scans) and the acquisition time was set at 2.60 s. All ¹H-NMR chemical shifts reported are relative to the peak of CHCl₃ set at 7.26 ppm. A quantitative determination of the functional groups was obtained by ³¹P-NMR using a 400 MHz

Table 1. Experimental conditions for suberin deconstruction with ethanolamine and partial substitution with glycerol. Data reported for recovery yield (% cellulose rich and extractives) were the means of two experiments. All the experiments were performed starting from 0.5 g of destarched potato peel.

	Time (min)	Temperature (°C)	Consistency (w v ⁻¹)	Glycerol (mL)	Ethanolamine (mL)	Cellulose rich (%)	Extractives (%)
A	120	120	10	0.00	5.00	82 ± 3	18 ± 3
B	120	120	10	4.00	1.00	85 ± 2	18 ± 1
C	120	120	10	4.25	0.75	84 ± 2	15 ± 2
D	120	120	10	4.50	0.50	88 ± 3	13 ± 2
E	120	120	10	4.75	0.25	94 ± 1	7 ± 2
F	120	120	10	5.00	0.00	98 ± 1	3 ± 1

Bruker Avance NMR spectrometer. Dried extractives (ca. 20 mg) were dissolved in a mixture of pyridine-deuterated chloroform (1.6:1 v v⁻¹, 700 μ L) containing 1 mg mL⁻¹ of chromium(III) acetylacetonate (Cr(acac)₃). Solutions of endo-*N*-hydroxy-5-norbornene-2,3-dicarboximide (100 μ L, 121.5 mmol L⁻¹, pyridine/CDCl₃ 1.6:1 v v⁻¹) and 2-chloro-4,4,5,5-tetramethyl-1,3,2-dioxaphospholane (100 μ L) were added as internal standard and derivatizing agent, respectively. The reported values are the means of two replicates with percentage errors generally <5% relative to the mean. Gas chromatography–mass spectrometry (GC–MS) analyses were performed injecting 2 μ L of sample (0.1 μ g mL⁻¹, hexane GC grade) into a gas chromatograph (HP 5890 series II)/electron impact mass spectrometer (HP 5972 series) equipped with a HP-5MS column capillary (30 m length, 0.251 mm inner diameter, 0.25 μ m film thickness, 95% dimethylpolysiloxane). The injection was in a splitless mode with a temperature of 280 °C. The column was eluted at 60 °C for 5 min, followed by a temperature gradient from 60 to 280 °C at 8 °C min⁻¹, and 15 min at 280 °C. The gas carrier was helium (99.9999% purity) at a pressure of 10 psi (1 mL min⁻¹). For identification, each peak (area > 1%) shown in GC–MS chromatogram was matched with the National Institute of Standards and Technology (NIST) mass spectral library, LIPID MAPS (<https://www.lipidmaps.org/>) and the relevant literature.²⁰

Results and discussion

Ethanolamine is a dual-functional solvent, serving as both a hydrogen bond donor and acceptor via its hydroxyl (-OH) groups, making it an effective organo-solvent for biorefinery

applications.¹² Its amine (-NH₂) group also enables it to act as a proton/hydrogen ion acceptor, enhancing its reactivity as a base and/or nucleophile in biomass deconstruction.¹³ In order to reduce the ethanolamine consumption, glycerol was used as a low-cost, low-toxicity, high-boiling point co-solvent. Water is commonly studied as a co-solvent with alkanolamines,¹² but must be avoided to achieve selectivity for amides. After the treatment, in order to recover the purified fractions, we used ethanol to wash out the ethanolamides, and then water to remove glycerol and unreacted ethanolamine from the cellulose-rich solids. The conditions (time, temperature, and consistency) and the percentage gravimetric yields (cellulose rich and extractives) of the experiment performed in pure ethanolamine, in mixtures of glycerol and ethanolamine, and in pure glycerol as reference are reported in Table 1. With pure ethanolamine (A) treatment, the processing of potato peels yielded two fractions of 82% (cellulose rich) and 18% (extractives), whereas only 3% of extractives were recovered from the pure glycerol treatment (F). In order to reduce ethanolamine, a series of experiments was performed at 20% (B), 15% (C), 10% (D), and 5% (E) of ethanolamine in glycerol. At 15 and 20% of ethanolamine in glycerol, similar yields to pure EA were obtained, while at 10% and 5% the yields were lower.

Figure 1 shows the FTIR spectra of all recovered materials, with key bands highlighted to elucidate the chemical effects of the treatment. In the left panel, representing cellulose-rich materials, the bands at 1740, 1645, 1610, and 1508 cm⁻¹ – associated with suberin (aliphatic esters and aromatic lignin domains) – were significantly reduced in intensity following treatment with ethanolamine (EA). A similar reduction was

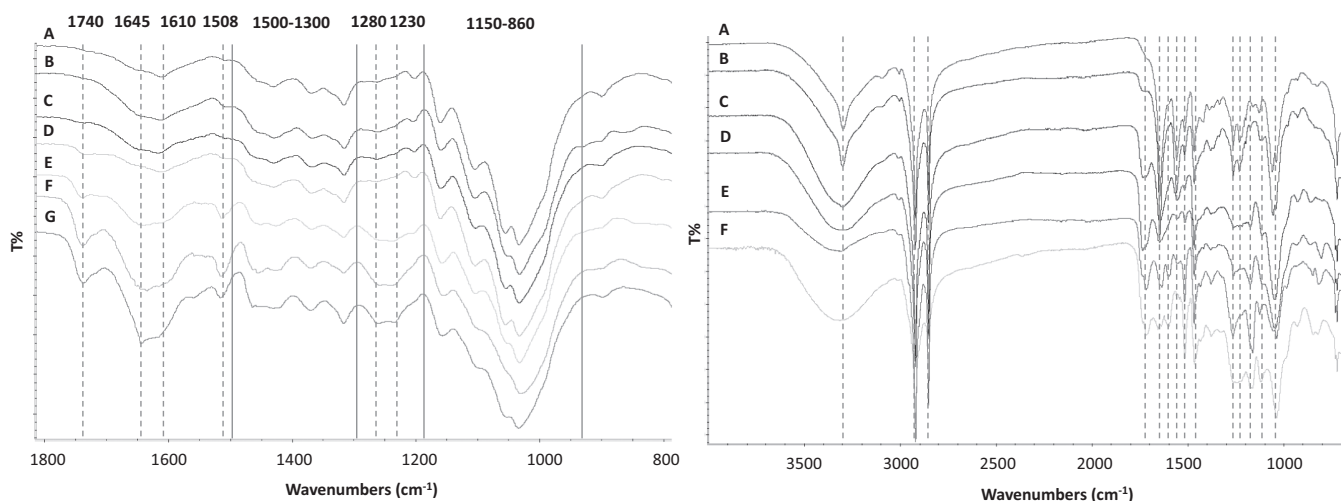


Figure 1. Fourier transform infrared stacked spectra of cellulose rich material (left) and extractives (right) recovered at different ethanolamine concentration (Table 1). Dot lines and solid lines highlight relevant infrared bands and intervals. The spectrum G represents the destarched potato peel.

observed for the bands at 1280 and 1230 cm^{-1} . In contrast, the regions between 1500–1300 cm^{-1} and 1150–900 cm^{-1} , attributed to polysaccharides, remained unaffected. A clear trend was observed with increasing ethanolamine concentration in the mixture.

A similar pattern was noted in the spectra of extractives (Figure 1, right panel). In the spectrum corresponding to pure glycerol treatment (spectrum F), extractives exhibited bands characteristic of aliphatic esters and aromatic compounds. As ethanolamine concentration increased, the intensity of bands corresponding to ethanolamides also increased. Notably, bands at 3300 (N–H stretch), 2930 and 2870 (C–H stretch), 1645 and 1560 (amide I and II), 1480 and 1230 ($-\text{CH}_2-$), and 1050 cm^{-1} (C–O alcohol) became more prominent. The ester- and lignin-related bands at 1508 and 1260 cm^{-1} disappeared.

The reactions involved in suberin deconstruction were interpreted as nucleophilic acyl substitution ($\text{S}_{\text{N}}\text{Ac}$), in particular as an aminolysis of ester bonds, resulting in the formation of ethanolamides and alcohols (Fig. 2). The extractives from experiment B were characterized using NMR techniques (Fig. 3). The general structure of ethanolamides was evident in the ^1H -NMR spectrum (Fig. 3, left panel). In addition to peaks corresponding to alkyl chains (primarily unsaturated), characteristic multiplet peaks for ethanolamides were observed at 3.43 and 3.72 ppm.¹⁸ Other relevant signals indicated the presence of aliphatic alcohols, with a triplet at 3.64 ppm.

The phosphorus-31 nuclear magnetic resonance (^{31}P -NMR) analysis (Fig. 3, right panel), following derivatization, enabled quantification of various functional groups in the extractives.²¹ Specifically, the extractives were

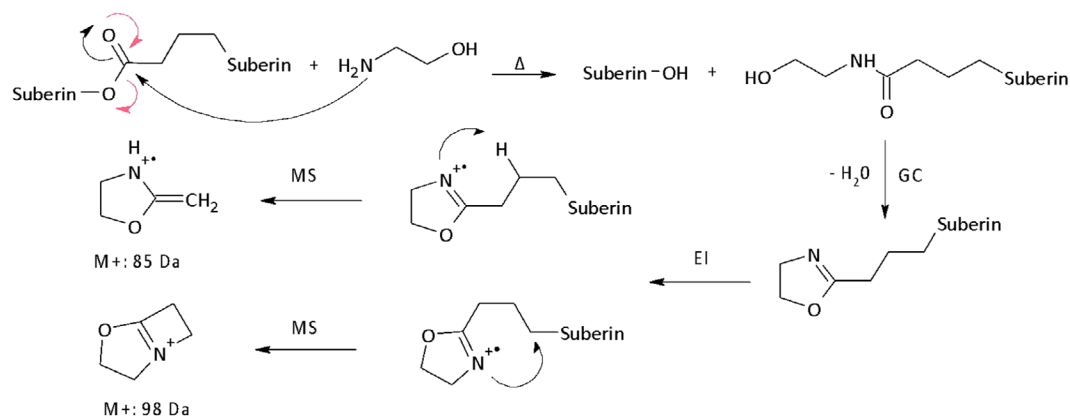


Figure 2. Chemical transformation during the ethanolamine deconstruction of suberin contained in potato peel and gas chromatography–mass spectrometry (GC–MS) analyses. (1) nucleophilic acyl substitution ($\text{S}_{\text{N}}\text{Ac}$)-aminolysis of ester by ethanolamine attack leading to ethanolamides and alcohols; (2) dehydration reaction of ethanolamines in the GC injector to 2-oxazoline derivatives; (3) the main 2-oxazoline radical cation (generated in electron ionization, EI) fragmentation pathway to the base ion peaks at 98 and 85 Da.

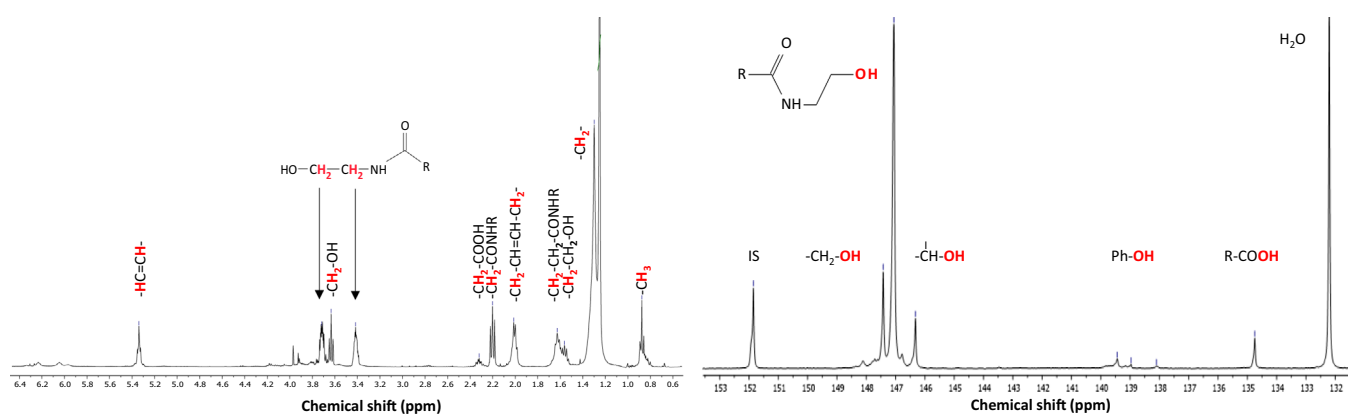


Figure 3. Proton nuclear magnetic resonance (^1H -NMR) (left) and phosphorus-31 nuclear magnetic resonance (^{31}P -NMR) (right) spectra of extractives from potato peel obtained in experiment B. Attributions are highlighted in red for proton in ^1H -NMR and labile proton groups in ^{31}P -NMR.

rich in ethanolamides (3.44 mmol g⁻¹), with additional functionalities including primary alcohols (0.90 mmol g⁻¹), secondary alcohols (0.41 mmol g⁻¹), and phenols (0.29 mmol g⁻¹), confirming the ¹H-NMR findings. Carboxylic acids were present in only trace amounts (0.19 mmol g⁻¹).

A more detailed characterization was performed by GC-MS (Supporting Information, Fig. S1). As reported in the literature, ethanolamides have a strong tendency to dehydrate and form 2-oxazolines during the GC injection (220 °C).²² The main fragmentation of oxazolines is illustrated in Fig. 2, and the fragments at 85 and 98 Da were considered diagnostic for oxazoline identification. Table 2 reports the number of peaks, retention times (RT, min), area percentages (%), and attributions (Supporting Information, Figs S2–S12).²³

The chemical structures of the 24 chromatographic peaks were identified (excluding peaks 20 and 23). The extractives from experiment B consisted primarily of aliphatic alcohols (13.1%) with chain lengths exceeding 16 carbon atoms, and oxazoline derivatives (66.6%) formed by dehydration of ethanolamides. A small amount of aromatic compounds (4.5%) was detected, which was unequivocally attributed to vanillin. These attributions and quantities align well with the literature on suberin composition.²⁴ The two major compounds (peaks 15 and 21), which accounted for nearly 50% of the sample, were identified as (9Z)-N¹,N¹⁸-bis(2-hydroxyethyl)-9-octadecenediamide and (9Z)-N-(2-hydroxyethyl)-18-hydroxy-9-octadecenamamide, derivatives of the main acids of suberin (octadecenedioic and 18-hydroxyoctadecenoic acids) after reaction with ethanolamine.²⁰

Conclusions

The ethanolamine-based reactive organosolv processing of potato peels yielded two notable fractions: an 80% cellulose-rich material and nearly 20% ethanolamides. The valorization of cellulose-rich material was well established, but ethanolamides represented an innovative biobased raw material. Their structural motifs suggested diverse applications, including (i) as diols (due to the particular structure of suberin fatty acids) in polyesters and polyurethanes,¹⁸ with the long-chain diol nature of the extractives providing a low glass transition temperature for specific applications and exploiting the amide bonds for biodegradability,²⁵ (ii) as precursors for bis-2-oxazoline derivatives,²³ formed via ethanolamide ring closure and widely used as polymer chain extenders,²⁶ (iii) as foam-boosting agents and/or surfactants in cosmetics,¹⁶ with

Table 2. Gas chromatograms (reported in Supporting Information, Fig. S1), peak retention time (min), area (%) and attributions of extractives B by electron ionization (EI) mass spectrum interpretation.

	RT (min)	AREA (%)	Attribution	Notes
1	16.92	4.5	Aromatic	Vanillin
2	19.47	1.3	Aliphatic OH	C16
3	21.96	2.2	Aliphatic OH	C18
4	24.21	2.0	Aliphatic OH	C20
5	25.79	1.6	Ethanolamide	From C12 dioic
6	26.25	1.2	Aliphatic OH	C>20
7	27.50	1.5	Aliphatic OH	C>20
8	27.78	1.2	Ethanolamide	From C18== (linoleic)
9	27.85	1.0	Ethanolamide	From C18=(oleic)
10	28.13	1.0	Ethanolamide	From C14 dioic
11	28.37	1.8	Oleamide	C>20
12	28.67	1.7	Aliphatic OH	C>20
13	30.17	2.0	Aliphatic OH	C>20
14	30.60	1.7	Ethanolamide	Not oxazoline C18=OH
15	32.40	29.0	Ethanolamide	From C18=OH
16	33.90	1.2	Aliphatic OH	C>20
17	34.45	3.7	Ethanolamide	?
18	35.38	1.2	Ethanolamide	From C18 dioic
19	36.67	3.2	Ethanolamide	?
20	37.68	2.7	Unknown	
21	39.35	18.0	Ethanolamide	From C18= dioic
22	41.91	3.0	Ethanolamide	From C22 dioic
23	45.91	2.0	Unknown	
24	52.16	2.0	Ethanolamide	From C24 dioic
		90.7	Total	
		66.6	Ethanolamides	
		13.1	Aliphatics OH	
		4.5	Aromatics	

palmitoyl ethanolamide as a technological reference; and (iv) as bioactive materials,^{17,19} given that naturally occurring ethanolamides function as lipid neurotransmitters and autacoids, including an anti-inflammatory ethanolamide derivative of azelaic acid.²⁷

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