Journal of Chromatography Open 9 (2026) 100301

Contents lists available at ScienceDirect

Journal of Chromatography Open

journal homepage: www.elsevier.com/locate/jcoa

Preliminary characterization of wind turbine blade waste by solvolysis, microwave-assisted extraction, and comprehensive two-dimensional gas chromatography coupled with time-of-flight mass spectrometry

Giulia Giacoppo [a,c], Charlotte Mase [b,c], Marco Piparo [b,c], Pierre Giusti [b,c], Caroline Mangote [b,c], Callum Branfoot [d], Luisa Pasti [e], Flavio Antonio Franchina [a,c,*], Marco Beccaria [a,c,f,*]

[a] Department of Chemical, Pharmaceutical and Agricultural Sciences (DOCPAS), University of Ferrara, Ferrara, Italy
[b] TotalEnergies OneTech, TotalEnergies Research & Technology Gonfreville, Harfleur BP27 76700, France
[c] International Joint Laboratory – iC2MC: Complex Matrices Molecular Characterization, TRTG, Harfleur, France
[d] Engineering Operations, NCC, Feynman Way, Emersons Green, Bristol BS16 7FS, United Kingdom
[e] Department of Environmental and Prevention Sciences, University of Ferrara, Ferrara, Italy
[f] TotalEnergies OneTech, Zone industrielle C, 7181 Seneffe, Belgium

ARTICLE INFO

Keywords:
Wind turbine blades
Solvolysis
Microwave-assisted extraction
GC×GC-TOFMS
New energy materials

ABSTRACT

The growth of renewable energy infrastructure highlights the urgent need for sustainable solutions to manage its end-of-life materials, including wind turbine blades (WTBs). These composite materials present significant challenges in recycling due to their complex heterogeneous structure and varied chemical composition. To depolymerize WTB composite materials, a solvolysis process was applied. The resulting chemical products underwent microwave-assisted extraction (MAE), using a mixture of *n*-hexane and methanol (10:3, v:v). The non-polar organic phase was subjected to analysis using comprehensive two-dimensional gas chromatography–time-of-flight mass spectrometry (GC×GC-ToFMS). On the basis of electron impact ionization mass spectral matching at 70 eV, linear retention index data, and molecular distribution on the two-dimensional space, >110 compounds were putatively identified, mainly composed by aromatic and heteroatom-containing species. To the best of our knowledge, this is the first time that the molecular composition of WTBs has been investigated by MAE-GC×GC–MS.

1. Introduction

Wind energy plays a key role in Europe's transition to climate neutrality, as outlined in the European Green Deal, which aims for climate neutrality by 2050 [1,2]. However, many wind farms were built before circular economy principles were a priority, leading to challenges in managing end-of-life wind turbine blades (WTBs). Given their complex material composition, as well as the limited disposal options, decommissioned WTBs present a growing environmental concern. With waste generation expected to increase over the coming years, the development of sustainable recycling solutions is essential to align with the European Green Deal's objectives of clean energy and responsible resource management.

One of the most significant challenges in recycling technologies is efficiently recovering carbon and/or glass fibers from the polymeric matrix without compromising their mechanical properties [3]. Microwave-assisted methods, including chemical recycling and pyrolysis, are gaining attention for their ability to depolymerize composite matrices and facilitate fiber recovery [4]. For instance, Rani et al. [5] developed a sustainable microwave-assisted chemical recycling process using hydrogen peroxide and acetic acid under microwave irradiation to recycle glass fiber-reinforced polymer waste from wind turbine blades. However, in this paper, no investigation of the matrix recyclates were discussed. Similarly, Zabihi et al. [6] proposed a method using hydrogen peroxide combined with tartaric acid under microwave irradiation to decompose the epoxy matrix in glass fiber-reinforced composites. In this work, the authors recovered the decomposed polymer matrix residue, and reused it as a filler and reinforcement in new adhesive composites. Yet, no chemical characterization of the decomposed matrix was conducted. Also, a study by Åkesson et al. [7] explored the use of microwave

* Corresponding authors at: Department of Chemical, Pharmaceutical and Agricultural Sciences (DOCPAS), University of Ferrara, Ferrara, Italy.
E-mail addresses: flavioantonio.franchina@unife.it (F.A. Franchina), marco.beccaria@unife.it (M. Beccaria).

https://doi.org/10.1016/j.jcoa.2025.100301
Received 20 August 2025; Received in revised form 14 November 2025; Accepted 9 December 2025
Available online 9 December 2025

pyrolysis to recycle glass fiber-reinforced composites from decommissioned wind turbine blades, resulting in the recovery of glass fibers with a significant portion of their original mechanical properties. They further analyzed the residual coating on the recovered fibers by GC–MS, but no full characterization of the decomposed products was reported.

However, most of the existing studies primarily focus on the recovery and quality of fibers, with less emphasis on the detailed molecular characterization of the depolymerized matrix products [8,9]. Understanding the molecular composition is essential for optimizing recycling processes and valorizing by-products. Combining microwave-assisted extraction (MAE) with comprehensive two-dimensional gas chromatography (GC×GC) offers a powerful analytical approach to address this lack of information. Particularly considering the non-homogeneous nature of the samples, MAE can enhance the efficiency of extracting matrix degradation products, by improving solvent penetration, accelerating the extraction process, and enhancing overall extraction performance through the localized heating effects of microwave (MW) energy [10]. GC×GC provides significant advantages over conventional mono-dimensional (1D) GC, particularly when dealing with complex samples, by providing enhanced resolution, improved sensitivity and increased peak capacity [11]. This enables detailed characterization and the generation of structured chromatograms, which facilitate data interpretation.

To address this gap in the literature, a MAE-GC×GC–MS methodology was employed for the first time to characterize solvolysis products from wind turbine blades, to both identify value-added chemicals and potentially problematic molecules such as heteroatoms containing compounds. By providing detailed information on the molecular composition of depolymerized matrix products, this work aims to contribute to the development of more efficient and sustainable recycling strategies.

2. Materials and methods

2.1. Chemicals and sample treatment

A sample of shredded WTB was sourced by the NCC (United Kingdom) and identified as primarily a combination of glass fibres, unsaturated polyester resin, and balsa core material through indicative pyrolysis-GC–MS and FTIR experiments. A low temperature solvolysis adapted from the method of Protsenko and Petrov was devised and performed in a jacketed stainless-steel reactor with the following optimized conditions—160 °C, 1.25 M NaOH in mono-ethanolamine, 23 mg/mL *resin*/solvent ratio, 13 h, ambient pressure [12]. After the solvolysis reaction, the glass fibres were filtered and separated, leaving a combination of solvent, catalyst, and composite degradation products for subsequent analysis. Despite the separation of fibres, the provided solvolysis samples contained both a major liquid fraction and a minor solid fraction, which are shown in Fig. 1. The vial on the left (a) contains the liquid phase, while the vial on the right (b) contains the deposit phase. *n*-Hexane, methanol, *n*-alkanes mixture in a range from n-C7 to n-C30, and sodium sulfate were purchased from Merck KGaA (Darmstadt, Germany). Hydrochloric acid (34–37 %) was purchased from Carlo Erba Reagents S.r.l. (Milan, Italy).

2.2. Microwave-assisted extraction (MAE)

Microwave-assisted extraction (MAE) was performed using an Ethos UP microwave (MW) system equipped with a FastEX-12 rotor (Milestone Srl, Bergamo, Italia). Briefly, for both the liquid and the solid phase separately, approximately 1 g of sample was weighed and neutralized with hydrochloric acid. Next, 20 mL of an *n*-hexane/methanol mixture (10:3, v:v) was added, and the vessels were placed inside the MW oven. The MW conditions were as follows: the temperature ramped up to 80 °C in 2 min and was held for 10 min, with the MW power set to 1500 W. After cooling down to room temperature in approximately 20 min, the

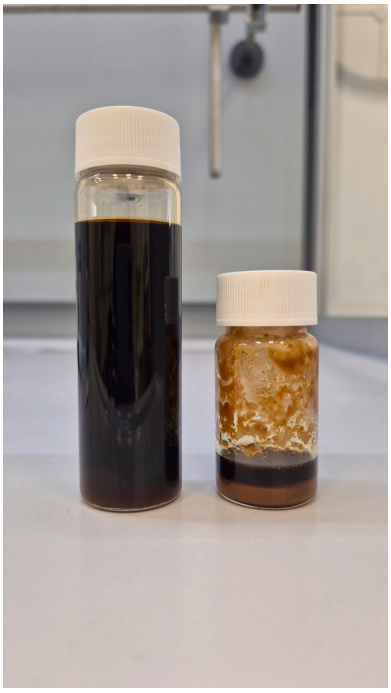

Fig. 1. Vials showing the products obtained from the solvolysis process: a) the liquid phase and b) the solid phase.

mixtures were transferred into a Falcon tube, and MQ water was added in a ratio of 2.5, followed by a centrifugation step for 20 min at 6000 rpm. After this step, the upper organic phase was collected and dried to a final volume of 1.5 mL for the following GC×GC analysis. Fig. 2 shows the workflow for sample preparation. The extraction procedure as well as the GC×GC–MS analysis was performed in triplicate. Additionally, a

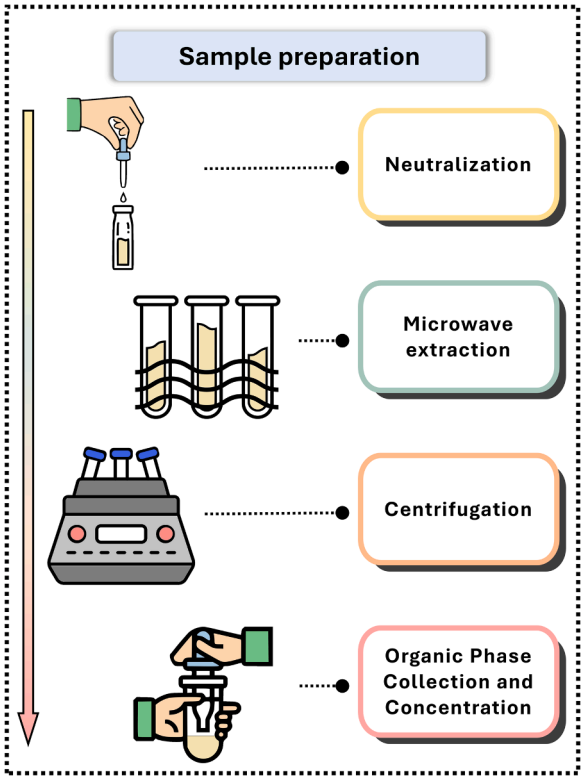

Fig. 2. Schematic representation of the sample preparation workflow.

blank extraction was also carried out.

2.3. GC×GC-ToFMS analysis

The GC×GC analyses were conducted on a Pegasus BT 4D (LECO Corporation, Mönchengladbach, Germany). The GC×GC column set consisted of a conventional (non-polar × polar) configuration. The first dimension (^{1}D) column was a non-polar Rxi-5MS (5 % diphenyl/95 % dimethyl polysiloxane, 30 m × 0.25 mm i.d. × 0.25 µm df, Restek Corp. Bellefonte, PA, USA), while ^{2}D column was a mid-polar Rxi-17Sil MS (50 % phenyl/50 % methyl polysiloxane with a silarylene group built into the backbone of the siloxane chain, 1.45 m × 0.25 mm i.d. × 0.25 µm df, Restek Corp. Bellefonte, PA, USA). The injections were performed in split mode (1:10), injection volume of 1 µL, inlet temperature of 300 °C. The carrier gas used was helium at 1.5 mL/min column flow throughout the run. The oven temperature program was 45 °C (held 1 min), then ramped at 6 °C/min to 335 °C. Temperature offsets for the secondary oven and for the modulator were set at +5 °C and +15 °C, respectively. The modulation period was set at 2 s. A time-of-flight mass analyzer was used to acquire a mass range from *m/z* 40 to 500 at 100 Hz using electron ionization (70 eV). The ion source was set at 250 °C, while the transfer line was set at 280 °C. An acquisition delay of 190 s was used. Data was collected and processed using ChromaTOF® BT software version 5.56.53 (LECO Corporation). The signal-to-noise threshold for peak integration was set at 100. Putative identification was based on the combination of the MS similarity with the NIST20 library (800/1000) with the confirmation using experimental linear retention index (LRI) within a ± 20 and the location of the molecules investigated on the 2D-GC plane.

3. Results and discussion

3.1. Extraction assisted by microwave

While MAE has been widely applied to extract compounds of interest in various matrices [13–15], its specific application to solvolysis products, to the best of the authors' knowledge, has never been explored. In this study, the potential of MAE was investigated in order to isolate value-added chemicals, while addressing compounds that may cause problems during the recycling process itself, from the products obtained after the solvolysis of WTBs. In fact, it is well reported in literature that some heteroatomic compounds are known to act as catalyst poisons and affecting the efficiency of upgrading processes, as well as creating problems of corrosion and thermal instability [16–20]. Hence, this highlights the essential role of sample preparation approaches in this field, in isolating and identifying potentially problematic species. Direct analysis of the solvolysis products was not feasible due to their extremely basic pH and the nature of the solvents used in the solvolysis process, which made direct injection into the GC×GC system unfeasible. Therefore, solid-liquid and liquid-liquid microwave-assisted extractions were performed using a mixture of two solvents of a different polarity, which were *n*-hexane and methanol, in the ratio 10:3. The selection of this solvent combination was based on critical considerations for both the MAE and subsequent GC×GC–MS analysis. In MAE, consideration should be given to the microwave-absorbing properties of the solvent, the interaction of the solvent with the matrix, and the analyte solubility in the solvent [10]. Since non-polar solvents such as *n*-hexane do not heat in a microwave field due to their low dielectric constant, *n*-hexane was combined with methanol, a polar solvent with high dielectric loss, to enable rapid and efficient heating of the extraction mixture. Moreover, the partial immiscibility of these two solvents under the extraction conditions enabled biphasic partitioning, allowing methanol, as a polar solvent, to facilitate the isolation of compounds which could interfere with GC analysis, while *n*-hexane, a non-polar aprotic solvent, selectively extracted analytes of interest which were directly compatible with the GC investigation.

3.2. GC×GC analysis

Using MAE-GC×GC-ToFMS methodology 115 molecules were putatively identified. The identification criteria were based on (i) mass spectral EI database matching at 70 eV (≥ 800/1000); ii) LRI window in the ± 20 range, based on the ^{1}D LRI information reported in the NIST database and iii) chemical class 2D plot position. For the LRI calculation, an *n*-alkanes solution (C_7—C_{30}) was employed. The analytes putatively identified belong to various chemical classes (Fig. 3), mainly composed of aromatics (25 %) heteroatoms-containing compounds, such as oxygen-containing compounds (50 %), nitrogen/oxygen-containing compounds (4 %) and nitrogen-containing compounds (16 %).

Among the chemical classes detected, hydrocarbons accounted for 4 % of the analytes. The chromatograms of the blanks revealed presence of branched hydrocarbons, indicating contamination likely introduced by the tubes used during the centrifugation step, which are made from polypropylene. For this reason, hydrocarbons detected in the sample that were also present in the blank were not taken into account. As a result, the reported hydrocarbon content represents only the portion of analytes which can be attributed to the sample, excluding any potential interference from the blank. As an example, Fig. 4 shows the blank-subtracted chromatograms of the fraction extracted from the liquid (Fig. 4a) and the solid phase (Fig. 4b). The non-subtracted chromatograms are provided in supplementary materials: figure S1 and figure S2 show the chromatogram of the fraction extracted from the liquid and the solid phase, respectively, while figure S3 shows the chromatogram of the blank.

A significant number of heteroatom-containing compounds, such as those containing oxygen and nitrogen were tentatively identified among the analytes. These compounds give rise to significant challenges in the recycling of WTBs, as their presence, particularly at high concentrations, can lead to issues such as corrosion, thermal instability, and catalyst poisoning during upgrading processes. These challenges are well-reported in literature on various samples both processed with different techniques, or before further processing [21–27], where several studies report how heteroatom-containing compounds contribute to instability, increased corrosiveness, and catalyst deactivation, overall, negatively impacting refining processes. This highlights the need for detailed characterization and appropriate pre-treatment before these recycled products can be successfully integrated into existing refinery systems.

A complete list of each compound tentatively identified is reported in Table 1.

The repeatability of the MAE-GC×GC–MS method was evaluated by conducting the extraction assisted by microwave in triplicate for both the liquid and solid samples, followed by GC×GC-MS analysis in triplicate for each. The results showed that the majority of the identified analytes exhibited a coefficient of variation below 15 %. Additionally, a one-way ANOVA was performed using a stringent significance level (α) of 0.001 to evaluate statistical differences among replicates. The analysis revealed that only approximately 10 % of the analytes were statistically different among replicates in both liquid and solid samples, making reliable the whole methodology. Figures S4 and S5 show the *p*-values on a negative logarithmic scale on the y-axis and the identified analytes on the x-axis, for the liquid and solid phases, respectively.

4. Limitation and future perspectives

This preliminary study focuses on the volatile fraction of the solvolysis product of WTBs. Future research should extend characterization efforts to include non-volatile fractions using advanced techniques such as FTICR-MS. One of the main limitations in the recycling of WTBs is the significant variation in their composition, which differs by manufacturer and model [28–30]. These variations directly impact the recyclability of WTBs, as a single recycling approach cannot be universally applied. Therefore, recycling methods must be adapted and optimized for different blade types, adding complexity to the development of

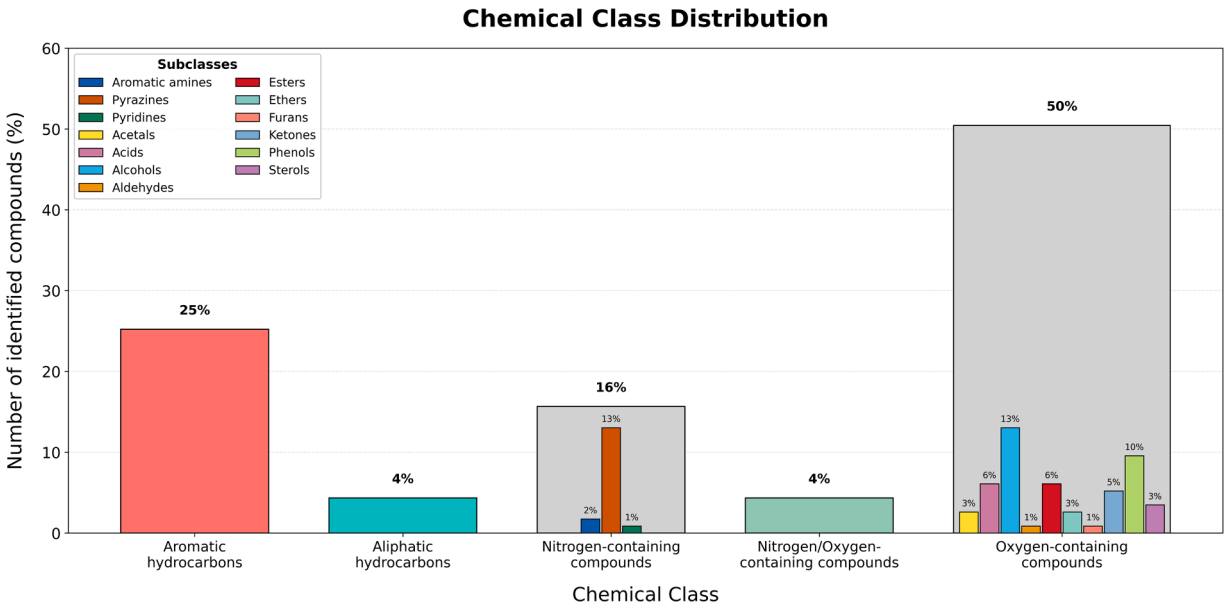

Fig. 3. Chemical class distribution on the basis of the number of identified compounds (%), considering both liquid and solid phases of solvolysis products from wind turbine blades.

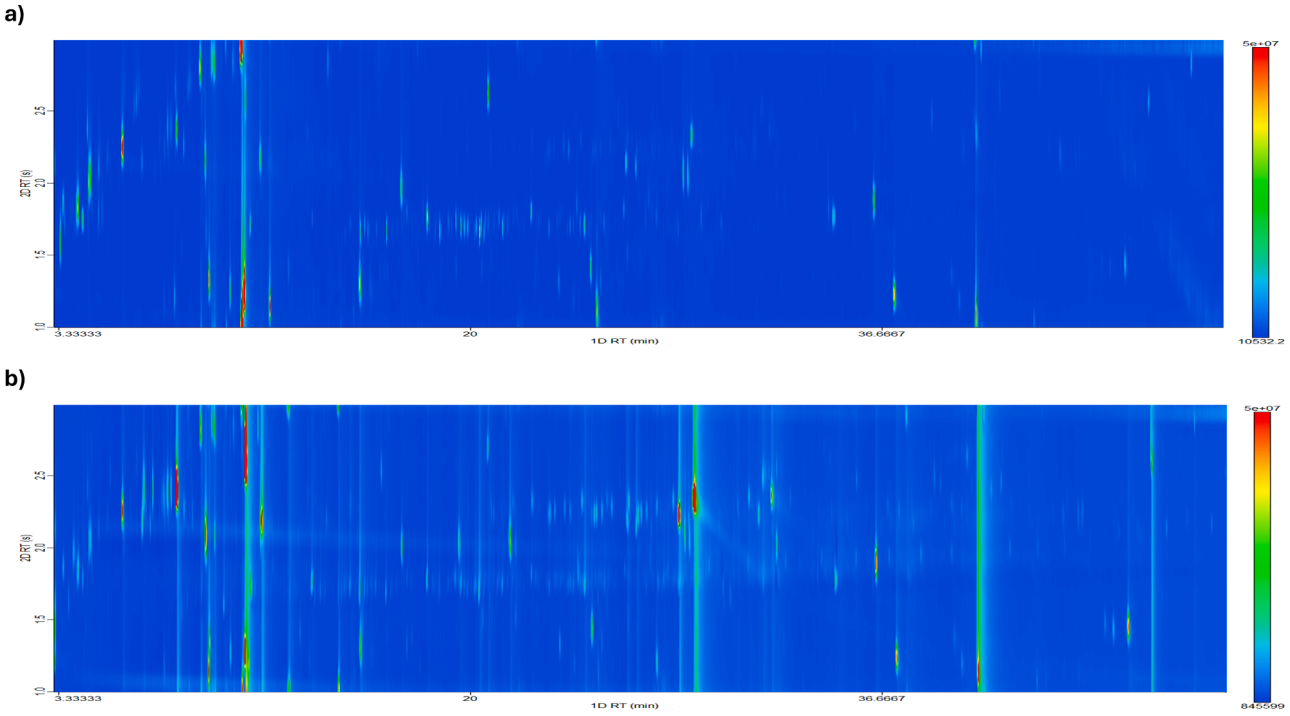

Fig. 4. Chromatograms of the liquid phase (a) and solid phase (b) with the blank subtracted.

standardized recycling solutions. According to the literature, solvolysis is a promising recycling technique due to its ability to break down composite materials while preserving the fibers with mechanical properties comparable to the virgin ones. Additionally, microwave-assisted processes are gaining attention as a promising alternative to enhance process feasibility [28]. However, these technologies still face challenges linked to scaling for industrial applications [8,31]. Future research should focus on refining these techniques, optimizing conditions for various WTB compositions, and addressing scalability issues to promote sustainable waste management within the renewable energy sector.

5. Conclusion

This study presents a comprehensive analytical approach for characterizing the products obtained from the solvolysis of WTBs, an area that has never been explored from an analytical point of view. A novel analytical approach was developed, including a first step of neutralization, followed by MAE, and finally GC×GC-ToFMS analysis, enabling a detailed characterization of solvolysis products. >110 compounds were tentatively identified, including various chemical classes such as aromatics and heteroatom-containing species. These findings have significant implications for recycling strategies: while aromatic compounds

Table 1
Compounds identified in liquid (LS) and solid (SS) samples. (OC: Oxygen-containing; NC: Nitrogen-containing; NOC: Nitrogen/Oxygen-containing; A: aromatic; HC: hydrocarbon). Note: compounds may belong to more than one category. For clarity, they are classified under their corresponding heteroatom-containing categories in this table.

Sample	Compound	Formula	Chemical Class	MS Similarity	Mean Exp RI	Lib. RI	^{1}D RT (min)	^{2}D RT (s)	CAS	S/N	CV %
SS	Glycolaldehyde dimethyl acetal	C4H10O3	OC	865	796		3.93	1.96	30,934–97–5	2947	11.18
LS/SS	1,4-Dioxane, 2,5-dimethyl-	C6H12O2	OC	915	804		4.1	1.83	15,176–21–3	3463/1969	3.41/9.86
LS/SS	Pyrazine, methyl-	C5H6N2	NC	960	822	829	4.47	2.4	109–08–0	1791/1416	5.95/12.72
SS	Ethanedioic acid, dimethyl ester	C4H6O4	OC	977	830	822	4.63	2.67	553–90–2	1121	10.95
LS/SS	2-Propanol, 1-(2-propenyloxy)-	C6H12O2	OC	936	844		4.91	2.1	21,460–36–6	1412/1004	7.18/14.17
LS	Styrene	C8H8	A	971	890	893	5.83	2.25	100–42–5	9165	6.89
LS	Cyclohexanone	C6H10O	OC	877	895	894	5.93	2.66	108–94–1	776	5.96
LS/SS	Ethanol, 2-butoxy-	C6H14O2	OC	936	907	907	6.2	2.08	111–76–2	206/213	19.41/21.47
LS	Pyrazine, 2,5-dimethyl-	C6H8N2	NC	971	911	917	6.3	2.53	123–32–0	902	6.91
LS/SS	Pyrazine, ethyl-	C6H8N2	NC	981	915	918	6.4	2.59	13,925–00–3	1507/1407	7.01/14.87
LS	Pyrazine, 2,3-dimethyl-	C6H8N2	NC	968	919	920	6.5	2.66	5910–89–4	966	7.71
LS	Pyridine, 2,4-dimethyl-	C7H9N	NC	884	936	931	6.9	2.62	108–47–4	158	16.81
LS	Pyridine, 2,3-dimethyl-	C7H9N	NC	885	948	943	7.18	2.73	583–61–9	165	12.06
LS	Aniline	C6H7N	NC	981	978	977	7.9	1.21	62–53–3	2854	5.42
LS/SS	Phenol	C6H6O	OC	971	979	981	7.93	2.67	108–95–2	1668/1672	11.17/19.81
LS/SS	α-Methylstyrene	C9H10	A	972	982	986	8	2.4	98–83–9	6521/14,167	7.43/6.98
LS/SS	Pyrazine, 2-ethyl-5-methyl-	C7H10N2	NC	978	1000	1005	8.43	2.67	13,360–64–0	1227/1046	7.93/15.64
LS/SS	Pyrazine, 2-ethyl-3-methyl-	C7H10N2	NC	957	1004	1004	8.53	2.72	15,707–23–0	815/584	7.35/16.24
LS	Pyrazine, 2-(n-propyl)-	C7H10N2	NC	927	1008	1010	8.63	2.74	18,138–03–9	241	8.79
LS	Pyridine, 2,3,6-trimethyl-	C8H11N	NC	913	1010	1003	8.68	2.64	1462–84–6	142	10.01
LS	2-Propanol, 1-(2-methoxypropoxy)-	C7H16O3	OC	918	1015	1010	8.8	2.44	13,429–07–7	386	6.29
LS/SS	2-Propanol, 1,1′-oxybis-	C6H14O3	OC	942	1019	1038	8.9	2.8	110–98–5	3764/4103	6.13/13.35
LS/SS	Benzene, 1,2,3-trimethyl-	C9H12	A	918	1023	1013	9	2.53	526–73–8	962/1336	5.73/12.25
LS/SS	Benzene, 1-propenyl-	C9H10	A	961	1026	1011	9.07	2.64	637–50–3	578/757	7.19/15.38
LS	1-Hexanol, 2-ethyl-	C8H18O	OC	953	1027	1030	9.11	2.15	104–76–7	2948	7.97
LS	Cyclohexane, butyl-	C10H20	HC	915	1031	1030	9.2	1.87	1678–93–9	236	5.36
SS	Butanedioic acid, dimethyl ester	C6H10O4	OC	982	1033	1032	9.24	1.11	106–65–0	5197	10.66
LS/SS	Benzyl alcohol	C7H8O	OC	957	1034	1036	9.27	1.32	100–51–6	3488/2987	7.54/12.69
LS/SS	1-Propanol, 2-(2-hydroxypropoxy)-	C6H14O3	OC	958	1038	1046	9.37	2.83	106–62–7	4799/3410	6.28/17.40
LS/SS	1-Propanol, 2,2′-oxybis-	C6H14O3	OC	943	1042	1042	9.47	2.8	108–61–2	3803/3340	6.33/11.67
SS	Benzaldehyde, 2-hydroxy-	C7H6O2	OC	924	1045	1047	9.53	1.24	90–02–8	177	16.55
LS	Phenol, 2-methyl-	C7H8O	OC	927	1053	1054	9.73	2.85	95–48–7	196	13.82
SS	Naphthalene, decahydro-, trans-	C10H18	HC	914	1055	1057	9.8	2.07	493–02–7	254	12.35
LS	Benzenemethanol, α-methyl-	C8H10O	OC	842	1059	1061	9.9	2.98	98–85–1	906	9.19
LS/SS	Acetophenone	C8H8O	OC	914	1067	1066	10.1	1.26	98–86–2	3292/3095	5.86/13.77
LS/SS	Dipropylene glycol (isomer 3)	C6H14O3	OC	907	1071	1068	10.2	2.85		1480/1329	6.24/13.26
SS	Branched Pyrazine	C8H12N2	NC	/	1084		10.5	2.71		228	14.54
LS/SS	Benzenemethanol, α,α-dimethyl-	C9H12O	OC	913	1086	1090	10.55	1.89	617–94–7	13,376/5086	5.25/11.93
LS	Pyrazine, 2,5-diethyl-	C8H12N2	NC	955	1089	1091	10.63	2.78	13,238–84–1	326	8.73
LS/SS	Phenol, 2-methoxy-	C7H8O2	OC	939	1090	1090	10.67	1.27	90–05–1	7377/10,330	4.36/10.13
SS	Benzene, (1-methoxy-1-methylethyl)-	C10H14O	OC	885	1092		10.7	2.63	935–67–1	39,420	5.70
SS	Benzaldehyde dimethyl acetal	C9H12O2	OC	928	1111		11.17	2.82	1125–88–8	3590	11.68
SS	Hexanoic acid, 2-ethyl-	C8H16O2	OC	960	1118	1123	11.34	2.17	149–57–5	6662	12.20
LS	Benzenamine, N-ethyl-	C8H11N	NC	933	1131	1128	11.67	1.14	103–69–5	5902	7.04
LS/SS	Cyclohexane, pentyl-	C11H22	HC	914	1135	1135	11.76	1.86	4292–92–6	436/715	5.09/11.19
LS	Phenol, 2-ethyl-	C8H10O	OC	879	1137	1140	11.8	2.88	90–00–6	114	11.34
LS	5H-5-Methyl-6,7-dihydrocyclopentapyrazine	C8H10N2	NC	899	1142	1144	11.93	1.24	23,747–48–0	191	9.71
LS	(*S*)-(+)-6-Methyl-1-octanol	C9H20O	OC	910	1144	1144	11.97	2.14	110,453–78–6	239	9.33

(*continued on next page*)

Table 1 (*continued*)

Sample	Compound	Formula	Chemical Class	MS Similarity	Mean Exp RI	Lib. RI	^{1}D RT (min)	^{2}D RT (s)	CAS	S/N	CV %
LS	Pyrazine, 3,5-diethyl-2-methyl-	C9H14N2	NC	911	1158	1162	12.3	2.62	18,138–05–1	393	7.53
LS	2,3,5-Trimethyl-6-ethylpyrazine	C9H14N2	NC	927	1161	1163	12.4	2.68	17,398–16–2	267	5.76
SS	Benzoic acid	C7H6O2	OC	935	1161	1177	12.4	1.45	65–85–0	4004	11.98
LS	2-Acetyl-3-ethylpyrazine	C8H10N2O	NOC	889	1167	1168	12.53	2.72	32,974–92–8	356	12.46
SS	1-Propanone, 1-phenyl-	C9H10O	OC	952	1167	1176	12.53	1.3	93–55–0	150	10.82
LS/SS	endo-Borneol	C10H18O	OC	869	1168	1167	12.57	2.61	507–70–0	304/257	7.38/ 12.03
LS	Benzeneethanol, β-methyl-	C9H12O	OC	863	1174	1179	12.71	1.24	1123–85–9	132	9.78
SS	Benzeneacetic acid, methyl ester	C9H10O2	OC	958	1179	1178	12.83	1.39	101–41–7	1284	11.39
LS	2-Methoxy-5-methylphenol	C8H10O2	OC	937	1181	1193	12.87	1.18	1195–09–1	739	9.25
LS	2-Decanone	C10H20O	OC	917	1192	1193	13.13	2.33	693–54–9	418	7.59
LS	α-Terpineol	C10H18O	OC	872	1192	1189	13.13	2.66	98–55–5	129	10.03
LS	Creosol	C8H10O2	OC	933	1195	1193	13.2	1.21	93–51–6	567	11.89
LS/SS	Quinoxaline	C8H6N2	NC	969	1211	1224	13.6	1.98	91–19–0	207/496	8.20/ 13.42
LS/SS	Furan, 3-phenyl-	C10H8O	OC	950	1224	1226	13.9	1.24	13,679–41–9	122/102	14.66/ 13.76
SS	Phenol, 3-(1-methylethyl)-	C9H12O	OC	934	1226	1228	13.93	2.91	618–45–1	686	13.04
LS	Cyclohexane, hexyl-	C12H24	HC	906	1239	1237	14.23	1.92	4292–75–5	236	9.29
SS	Hexanedioic acid, dimethyl ester	C8H14O4	OC	957	1244	1243	14.37	1.02	627–93–0	3408	12.94
LS	Benzoic acid, 2-propenyl ester	C10H10O2	OC	909	1255	1254	14.6	1.21	583–04–0	454	14.98
SS	1-Butanone, 1-phenyl-	C10H12O	OC	922	1255	1263	14.6	1.22	495–40–9	471	9.36
SS	1,1-Dimethoxy-2-phenylpropane	C11H16O2	OC	927	1262	1266	14.77	2.89	90–87–9	423	10.14
SS	Nonanoic acid	C9H18O2	OC	918	1264	1273	14.83	2.31	112–05–0	536	13.28
LS	Phenol, 2-methyl-5-(1-methylethyl)-	C10H14O	OC	900	1295	1299	15.54	2.93	499–75–2	234	8.25
SS	1,2-Benzenedicarboxylic acid	C8H6O4	OC	971	1317	1309	16.03	2.52	88–99–3	1844	9.49
LS/SS	Phenol, 2,6-dimethoxy-	C8H10O3	OC	946	1353	1355	16.83	1.96	91–10–1	3469/ 3321	6.42/ 11.60
LS	Diphenyl ether	C12H10O	OC	909	1405	1405	18	1.44	101–84–8	356	14.77
LS/SS	Phenol, 2-methoxy-4-(1-propenyl)-, (Z)-	C10H12O2	OC	888	1410	1408	18.1	1.3	5912–86–7	306/281	16.74/ 9.93
LS	1H-Isoindole-1,3(2H)-dione, 2-methyl-	C9H7NO2	NOC	947	1425	1425	18.4	2.43	550–44–7	471	6.83
LS/SS	5,9-Undecadien-2-one, 6,10-dimethyl-, (E)-	C13H22O	OC	922	1453	1453	19	2.56	3796–70–1	120/184	7.69/ 10.67
LS	Phenol, 2-methoxy-4-(1-propenyl)-	C10H12O2	OC	956	1453	1450	19	1.4	97–54–1	803	7.45
SS	Dimethyl phthalate	C10H10O4	OC	968	1457	1454	19.09	2.01	131–11–3	5414	11.39
LS/SS	Bibenzyl	C14H14	A	961	1524	1525	20.47	1.4	103–29–7	191/216	5.36/ 11.56
LS/SS	Benzene, (1-butylhexyl)-	C16H26	A	954	1538	1535	20.74	2.24	4537–11–5	248/642	10.41/ 7.90
LS/SS	Benzene, (1-propylheptyl)-	C16H26	A	931	1547	1546	20.93	2.24	4537–12–6	217/558	8.72/ 7.69
SS	Methyl hydrogen phthalate	C9H8O4	OC	942	1556	1561	21.1	2.02	4376–18–5	3925	11.71
SS	Dodecanoic acid	C12H24O2	OC	904	1562	1567	21.21	2.29	143–07–7	721	8.52
LS/SS	Benzene, (1-ethyloctyl)-	C16H26	A	938	1565	1568	21.28	2.27	4621–36–7	185/537	4.18/ 6.93
LS/SS	Benzene, (1-methylnonyl)-	C16H26	A	902	1601	1616	22	2.3	4537–13–7	506/ 1108	6.72/ 7.91
LS/SS	Benzene, (1-pentylhexyl)-	C17H28	A	945	1633	1628	22.6	2.22	4537–14–8	458/ 1525	6.00/ 16.08
LS/SS	Benzene, (1-butylheptyl)-	C17H28	A	952	1636	1632	22.66	2.24	4537–15–9	851/ 2668	6.16/ 5.59
LS/SS	Benzene, (1-propyloctyl)-	C17H28	A	947	1646	1643	22.85	2.24	4536–86–1	720/ 2098	4.49/ 6.23
LS/SS	Benzene, (1-ethylnonyl)-	C17H28	A	940	1667	1670	23.23	2.27	4536–87–2	566/ 1741	4.05/ 6.50
LS/SS	Benzene, (1-methyldecyl)-	C17H28	A	914	1704	1708	23.93	2.31	4536–88–3	1331/ 3876	4.54/ 7.28
LS/SS	Benzene, (1-pentylheptyl)-	C18H30	A	964	1731	1726	24.4	2.22	2719–62–2	704/ 2626	6.49/ 6.79
LS/SS	Benzene, (1-butyloctyl)-	C18H30	A	969	1736	1730	24.5	2.23	2719–63–3	755/ 2613	3.20/ 5.12
LS	1H-Isoindole-1,3(2H)-dione, 2-(2-hydroxyethyl)-	C10H9NO3	NOC	947	1738		24.54	1.12	3891–07–4	4767	6.46
LS/SS	Benzene, (1-propylnonyl)-	C18H30	A	957	1747	1742	24.7	2.24	2719–64–4	596/ 2246	5.71/ 9.70
LS/SS	Benzene, (1-ethyldecyl)-	C18H30	A	953	1770	1766	25.1	2.27	2400–00–2	494/ 1583	4.18/ 6.66
LS	(3-Methylbutane-1,3-diyl) dibenzene	C17H20	A	918	1772	1767	25.13	1.18	1520–43–0	163	14.97
LS	Anthracene	C14H10	A	956	1787	1786	25.4	2.47	120–12–7	1215	5.55

(*continued on next page*)

Table 1 (*continued*)

Sample	Compound	Formula	Chemical Class	MS Similarity	Mean Exp RI	Lib. RI	^{1}D RT (min)	^{2}D RT (s)	CAS	S/N	CV %
LS/SS	Benzene, (1-methylundecyl)-	C18H30	A	932	1808	1808	25.77	2.31	2719–61–1	1196/ 3820	4.94/ 9.58
LS/SS	Benzene, (1-pentyloctyl)-	C19H32	A	950	1830	1819	26.13	2.21	4534–49–0	713/ 3458	2.62/ 7.49
LS/SS	Benzene, (1-butylnonyl)-	C19H32	A	946	1836	1826	26.24	2.23	4534–50–3	477/ 2264	3.46/ 5.27
LS/SS	Benzene, (1-propyldecyl)-	C19H32	A	934	1849	1840	26.47	2.25	4534–51–4	369/ 1846	5.93/ 6.84
LS/SS	Benzene, (1-ethylundecyl)-	C19H32	A	937	1873	1865	26.87	2.27	4534–52–5	296/ 1403	4.99/ 6.84
LS/SS	Benzene, (1-methyldodecyl)-	C19H32	A	938	1911	1916	27.5	2.32	4534–53–6	812/ 3411	5.77/ 5.41
SS	Hexadecanoic acid, methyl ester	C17H34O2	OC	951	1925	1926	27.73	2.22	112–39–0	11,050	5.21
SS	n-Hexadecanoic acid	C16H32O2	OC	946	1963	1968	28.34	2.33	57–10–3	4297	8.67
LS/SS	Naphthalene, 2-phenyl-	C16H12	A	931	1981	1978	28.63	2.36	612–94–2	643/800	7.12/ 8.43
LS/SS	Oxazole, 4,5-dihydro-2-pentadecyl-	C18H35NO	NOC	887	2140	2137	31.06	2.55	36,919–66–1	703/866	1.82/ 6.42
LS/SS	m-Terphenyl	C18H14	A	884	2158	2158	31.33	2.28	92–06–8	224/264	4.37/ 9.55
LS/SS	Cyclohexane, 1,3,5-triphenyl-	C24H24	A	902	2458	2464	35.46	1.9	28,336–57–4	8582/ 14,284	9.56/ 6.51
LS/SS	Palmidrol	C18H37NO2	NOC	806	2523	2535	36.3	1.22	544–31–0	5500/ 7466	3.18/ 6.00
SS	Supraene	C30H50	HC	946	2833	2826	40	2.39	7683–64–9	517	2.53
SS	5-Cholestene-3-ol, 24-methyl-	C28H48O	OC	866	3241		44.46	1.47	290,299–12–6	183	5.93
LS/SS	Stigmasterol	C29H48O	OC	881	3272	3249	44.8	1.43	83–48–7	113/476	13.59/ 4.39
LS/SS	γ-Sitosterol	C29H50O	OC	899	3327	3321	45.4	1.44	83–47–6	401/ 1387	15.36/ 6.86
SS	Stigmasta-5,24(28)-dien-3-ol, (3β,24Z)-	C29H48O	OC	785	3342	3343	45.56	1.57	481–14–1	101	23.81

hold potential for valorization, the presence of heteroatoms poses challenges, including corrosion and catalyst deactivation during upgrading processes. The repeatability of the developed MAE-GC×GC-ToFMS method was also tested showing consistency in the results, with the majority of the analytes having a coefficient of variation below 15 %. In addition, one-way ANOVA was performed to test the repeatability of the entire MAE-GC×GC–MS procedure, with only about 10 % of the analytes showing statistically significant differences among replicates. The presented work contributes into the field of WTB recycling by providing preliminary chemical characterization of solvolysis products. These results align with the goals of the European Green Deal by supporting sustainable waste management and promoting circular economic practices.

CRediT authorship contribution statement

Giulia Giacoppo: Writing – review & editing, Writing – original draft, Software, Investigation, Formal analysis, Data curation. **Charlotte Mase:** Writing – review & editing, Supervision, Software, Resources. **Marco Piparo:** Writing – review & editing, Supervision, Software, Resources. **Pierre Giusti:** Writing – review & editing, Supervision, Software, Resources. **Caroline Mangote:** Writing – review & editing, Supervision, Software, Resources. **Callum Branfoot:** Writing – review & editing, Resources, Investigation, Formal analysis. **Luisa Pasti:** Writing – review & editing, Supervision, Resources. **Flavio Antonio Franchina:** Writing – review & editing, Visualization, Supervision, Software, Resources, Methodology. **Marco Beccaria:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Software, Resources, Project administration, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgment

The authors thank SusWIND for providing the sample and LECO Corporation for their technical support.

Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.jcoa.2025.100301.

Data availability

Data will be made available on request.

References

[1] European Commission, Communication from the commission, "The European Green Deal," (n.d.). https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=celex:52019DC0640 (accessed January 13, 2025).
[2] T.S. Kathrin Graulich, Winfried Bulach, Johannes Betz, Peter Dolega, Christine Hermann, Andreas Manhart, Valentijn Bilsen, Federico Bley, Emma Watkins, Emerging waste streams – challenges and opportunities, (2021) 1–89.
[3] I. Julian, A. García-Jiménez, A. Aguado, C. Arenal, A. Calero, V. Campos, G. Escobar, A.M. López-Buendía, D. Romero, E. Verdejo, N. García-Polanco, Advances in the circularity of end-of-life fibre-reinforced polymers by microwave intensification, Chem. Eng. Process. Process Intensif. 178 (2022) 109015, https://doi.org/10.1016/J.CEP.2022.109015.
[4] M. Rani, P. Choudhary, V. Krishnan, S. Zafar, A review on recycling and reuse methods for carbon fiber/glass fiber composites waste from wind turbine blades, Compos. B Eng. 215 (2021) 108768, https://doi.org/10.1016/j.compositesb.2021.108768.
[5] M. Rani, P. Choudhary, V. Krishnan, S. Zafar, Development of sustainable microwave-based approach to recover glass fibers for wind turbine blades composite waste, Resour. Conserv. Recycl. 179 (2022) 106107, https://doi.org/10.1016/j.resconrec.2021.106107.

[6] O. Zabihi, M. Ahmadi, C. Liu, R. Mahmoodi, Q. Li, M. Naebe, Development of a low cost and green microwave assisted approach towards the circular carbon fibre composites, Compos. B Eng. 184 (2020) 107750, https://doi.org/10.1016/J.COMPOSITESB.2020.107750.
[7] D. Åkesson, Z. Foltynowicz, J. Christéen, M. Skrifvars, Microwave pyrolysis as a method of recycling glass fibre from used blades of wind turbines, J. Reinf. Plast. Compos. 31 (2012) 1136–1142, https://doi.org/10.1177/0731684412453512.
[8] G. Oliveux, L.O. Dandy, G.A. Leeke, Current status of recycling of fibre reinforced polymers: review of technologies, reuse and resulting properties, Prog. Mater. Sci. 72 (2015) 61–99, https://doi.org/10.1016/j.pmatsci.2015.01.004.
[9] C. Branfoot, H. Folkvord, M. Keith, G.A. Leeke, Recovery of chemical recyclates from fibre-reinforced composites: a review of progress, Polym. Degrad. Stab. 215 (2023) 110447, https://doi.org/10.1016/J.POLYMDEGRADSTAB.2023.110447.
[10] C. Sparr Eskilsson, E. Björklund, Analytical-scale microwave-assisted extraction, J. Chromatogr. A 902 (2000) 227–250, https://doi.org/10.1016/S0021-9673(00)00921-3.
[11] P.J. Marriott, S.T. Chin, B. Maikhunthod, H.G. Schmarr, S. Bieri, Multidimensional gas chromatography, TrAC Trends Anal. Chem. 34 (2012) 1–21, https://doi.org/10.1016/J.TRAC.2011.10.013.
[12] A.E. Protsenko, V.V. Petrov, Recycling of fiberglass fillers obtained from polymer composites based on an epoxy vinyl ester binder, Mech. Compos. Mater. 58 (2022) 537–544, https://doi.org/10.1007/s11029-022-10048-9.
[13] M. Llompart, M. Celeiro, T. Dagnac, Microwave-assisted extraction of pharmaceuticals, personal care products and industrial contaminants in the environment, TrAC - Trends Anal. Chem. 116 (2019) 136–150, https://doi.org/10.1016/j.trac.2019.04.029.
[14] D. Ferrara, M. Beccaria, C.E. Cordero, G. Purcaro, Microwave-assisted extraction in closed vessel in food analysis, J. Sep. Sci. 46 (2023) 2300390, https://doi.org/10.1002/jssc.202300390.
[15] L. Sanchez-Prado, C. Garcia-Jares, T. Dagnac, M. Llompart, Microwave-assisted extraction of emerging pollutants in environmental and biological samples before chromatographic determination, TrAC - Trends Anal. Chem. 71 (2015) 119–143, https://doi.org/10.1016/j.trac.2015.03.014.
[16] D. Zanella, M. Romagnoli, S. Malcangi, M. Beccaria, T. Chenet, C. De Luca, F. Testoni, L. Pasti, U. Visentini, G. Morini, A. Cavazzini, F.A. Franchina, The contribution of high-resolution GC separations in plastic recycling research, Anal. Bioanal. Chem. 415 (2023) 2343–2355, https://doi.org/10.1007/s00216-023-04519-8.
[17] G.H.C. Prado, Y. Rao, A. De Klerk, Nitrogen removal from oil: a review, Energ. Fuel. 31 (2017) 14–36, https://doi.org/10.1021/acs.energyfuels.6b02779.
[18] C.S. Creaser, F. Krokos, K.E. ÓNeill, M.J.C. Smith, P.G. McDowell, Selective chemical ionization of nitrogen and sulfur heterocycles in petroleum fractions by ion trap mass spectrometry, J. Am. Soc. Mass Spectrom. 4 (1993) 322–326, https://doi.org/10.1016/1044-0305(93)85054-2.
[19] Y.H. Chan, S.K. Loh, B.L.F. Chin, C.L. Yiin, B.S. How, K.W. Cheah, M.K. Wong, A.C. M. Loy, Y.L. Gwee, S.L.Y. Lo, S. Yusup, S.S. Lam, Fractionation and extraction of bio-oil for production of greener fuel and value-added chemicals: recent advances and future prospects, Chem. Eng. J. 397 (2020) 125406, https://doi.org/10.1016/j.cej.2020.125406.
[20] M. Beccaria, Y. Zou, P.H. Stefanuto, A.L.M. Siqueira, A. Maniquet, M. Piparo, P. Giusti, G. Purcaro, J.F. Focant, Deeper investigation of oxygen-containing compounds in oleaginous feedstock (animal fat) by preparative column chromatography and comprehensive two-dimensional gas chromatography coupled with high-resolution time-of-flight mass spectrometry, Talanta 238 (2022) 123019, https://doi.org/10.1016/j.talanta.2021.123019.
[21] M. Beccaria, A.L.M. Siqueira, A. Maniquet, P. Giusti, M. Piparo, P. Stefanuto, J. Focant, Advanced mono- and multi-dimensional gas chromatography–mass spectrometry techniques for oxygen-containing compound characterization in biomass and biofuel samples, J. Sep. Sci. 44 (2021) 115–134, https://doi.org/10.1002/jssc.202000907.
[22] J. Hertzog, C. Mase, M. Hubert-Roux, C. Afonso, P. Giusti, C. Barrère-Mangote, Characterization of heavy products from lignocellulosic biomass pyrolysis by chromatography and fourier transform mass spectrometry: a review, Energy & Fuels 35 (2021) 17979–18007, https://doi.org/10.1021/acs.energyfuels.1c02098.
[23] E. Lazzari, C. Mase, D.C. Dayton, S. Marceau, G. Purcaro, J.F. Focant, M. Piparo, P. Giusti, Exploring molecular composition of upgraded pyrolysis bio-oil using GC×GC-(EI/PI)-TOF MS with different column set-ups, J. Anal. Appl. Pyrolysis. 181 (2024) 106569, https://doi.org/10.1016/j.jaap.2024.106569.
[24] C. Mase, M. Hubert-Roux, C. Afonso, P. Giusti, Contribution of atmospheric pressure chemical ionization mass spectrometry for the characterization of bio-oils from lignocellulosic biomass: comparison with electrospray ionization and atmospheric pressure photoionization, J. Anal. Appl. Pyrolysis. 167 (2022) 105694, https://doi.org/10.1016/j.jaap.2022.105694.
[25] C. Mase, J.F. Maillard, S. Marcuz, M. Hubert-Roux, C. Afonso, P. Giusti, Contribution of LDI and MALDI for the characterization of a lignocellulosic-based pyrolysis bio-oil, J. Am. Soc. Mass Spectrom. 34 (2023) 1789–1797, https://doi.org/10.1021/jasms.3c00197.
[26] M.S. Talmadge, R.M. Baldwin, M.J. Biddy, R.L. McCormick, G.T. Beckham, G. A. Ferguson, S. Czernik, K.A. Magrini-Bair, T.D. Foust, P.D. Metelski, C. Hetrick, M. R. Nimlos, A perspective on oxygenated species in the refinery integration of pyrolysis oil, Green Chem. 16 (2014) 407–453, https://doi.org/10.1039/C3GC41951G.
[27] W. Zeb, M. Roosen, P. Knockaert, S. Janssens, D. Withoeck, M. Kusenberg, J. Hogie, P. Billen, S. Tavernier, K.M. Van Geem, S. De Meester, Purification and characterisation of post-consumer plastic pyrolysis oil fractionated by vacuum distillation, J. Clean. Prod. 416 (2023) 137881, https://doi.org/10.1016/J.JCLEPRO.2023.137881.
[28] A. Fresneda Cruz, C. Chaine, M.B. Figueirêdo, G. Murillo-Ciordia, A. Sanz-Martinez, I.A.J. Burillo, Potentials and limitations of microwave-assisted chemical recycling of fiber-reinforced composites from wind blades, Sustain. Energy Fuels. (2024), https://doi.org/10.1039/d4se00242c.
[29] K.W. Lund, E.S. Madsen, State-of-the-art value chain roadmap for sustainable end-of-life wind turbine blades, Renew. Sustain. Energy Rev. 192 (2024) 114234, https://doi.org/10.1016/j.rser.2023.114234.
[30] F. Spini, P. Bettini, End-of-life wind turbine blades: review on recycling strategies, Compos. B Eng. 275 (2024) 111290, https://doi.org/10.1016/J.COMPOSITESB.2024.111290.
[31] G. Arora, M.K. Singh, S. Zafar, S.M. Rangappa, S. Siengchin, Introduction to microwave heating and its applications in the composite industry, (2024) 1–35. https://doi.org/10.1007/978-981-97-2772-8_1.