



Tire wear particles concentrations in gully pot sediments

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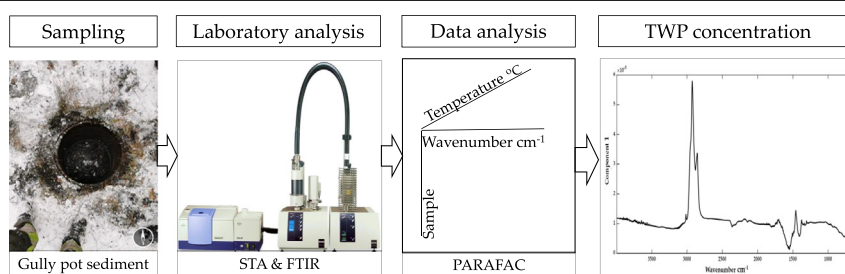
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HIGHLIGHTS

- Combining STA, FTIR and PARAFAC enables TWP quantification in environmental samples.
- No sample pretreatment is required and all particles sizes are included.
- TWP made up to 15% of gully pot sediments.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:

Received 17 October 2020

Received in revised form 19 December 2020

Accepted 21 December 2020

Available online 14 January 2021

Editor: Shuzhen Zhang

Keywords:

Road runoff

Tire wear particles

Thermogravimetry

FTIR

PARAFAC

ABSTRACT

While tire wear and tear is known to be a major source of microplastics in the environment, its monitoring is still hampered by the lack of analytical methods able to provide concentrations in environmental matrices. Tire wear particles (TWP) present in road runoff enter the drainage system through gully pots, built to prevent sediment deposition in the drainage system, and eventually protect downstream receiving waters. The aim of this study was to detect and quantify TWP in gully pot sediments, by using a novel method combining Simultaneous Thermal Analysis (STA), Fourier Transform Infrared (FTIR) spectroscopy and Parallel Factor Analysis (PARAFAC). The method was applied to samples from five sites in Southern Norway, characterized by different traffic densities and patterns. The method involved no sample pretreatment, the whole sediment sample was submitted to thermal decomposition in STA, and gases generated during pyrolysis were continuously transferred to FTIR. The FTIR data were arranged in a trilinear multi-way dataset (samples \times IR spectra wavenumber \times pyrolysis temperature) and then analyzed by PARAFAC. The results showed that TWP concentrations in gully pots varied greatly across sites, ranging from below 1 mg TWP/g sediment in streets with the lowest traffic densities, to 150 mg TWP/g sediment at the most trafficked study site. The results also indicated that other traffic conditions, such as driving patterns influence TWP concentrations. Finally, by enabling quantification of TWP in gully pot sediments, the approach presented here supports environmental monitoring of TWP and safe disposal of gully pot sediments, which is critical for environmental pollution management.

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1. Introduction

Road runoffs are found to contain substantial amounts of contaminants (Birch and Scollen 2003; Lindholm 2015) including tire wear particles (TWP) originating from vehicle tires (Andersson-Sköld et al. 2020). TWP is one of the major sources of microplastics in the

environment, and average emission per capita is estimated to be 0.81 kg/year worldwide and 0.9 kg/year in Norway (Kole et al. 2017; Vogelsang et al. 2018). In addition to potential adverse effects on human health, TWP could also affect aquatic and terrestrial ecosystems, where their main entrance way is through runoff (Kole et al. 2017; Wagner et al. 2018). Inflowing of TWP into water bodies begins with road runoff entering the drainage system through gully pots (Bolognesi et al. 2008). Gully pots are built to retain solids in the runoff and prevent or reduce sediment deposition in the drainage system and

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eventually protect the downstream receiving waters (Bolognesi et al. 2008; Karlsson and Viklander 2008; Lindholm 2015). Accumulation of TWP in gully pots is influenced by road deposition, particle size and density, stormwater inflow intensity (e.g. mobilization) and design of the gully pots (Bolognesi et al. 2008). Earlier studies (Kole et al. 2017; Panko et al. 2013) estimated that the major part of generated TWP would be coarse in size ($>10\ \mu\text{m}$), and deposited on or close to the road. This presents a high potential for TWP entering gully pots. However, the spread of TWP in the environment and the properties governing spreading processes are not adequately studied (Wagner et al. 2018). For example, the density of TWP is reported to be in the range of 1.13 to 1.16 g/cm^3 (Rhodes et al. 2012), while tires' main constituent part, carbon black (ca. 40%) (Roy et al. 2004), has a density of 1.8 g/cm^3 and styrene butadiene rubber (SBR) has a density of 0.94 g/cm^3 (CAS 2020). The lower densities of TWP compared to other road derived particles (e.g. asphalt 2.36 g/cm^3 , quartz 2.65 g/cm^3 or limestone 2.0 g/cm^3 (CAS 2020; Rumble, 2017)) suggest that TWP have a high potential to be transported over longer distances. However, studies reported that TWP aggregate with higher density particles in the road environment due to heat and friction created during driving (Kreider et al. 2010; Järtskog et al. 2020; Kole et al. 2017; Sommer et al. 2018; Wagner et al. 2018). This heteroaggregation would increase density and subject TWP to sedimentation.

The solids retained in gully pots build up overtime, necessitating a regular emptying to avoid clogging of the drainage system (Bolognesi et al. 2008). For safe disposal of gully pot sediments, it is critical to be able to quantify the environmental contaminants they may contain, including TWP. However, TWP monitoring in gully pots remains a challenge, mainly due to the lack of widely applicable, easy and inexpensive analytical methods (Järtskog et al. 2020).

While commonly used in microplastics studies, vibrational spectroscopic methods, such as Fourier transform infrared (FTIR) spectroscopy, have been found inapt for the analysis of TWP (Eisentraut et al. 2018; Wagner et al. 2018). Although attenuated total reflectance (ATR)-FTIR could be used for larger ($>0.5\ \text{mm}$) TWP particles (Fernández-Berridi et al. 2006; Manohar et al. 2017), the requirements of the methods (e.g. placing each isolated particle onto a glass slide, to bring it in close contact with the germanium crystal of the ATR objective), make its application challenging for TWP under 0.5 mm. In addition, although μ -FTIR methods use polymer specific absorption patterns of irradiated infrared and present unambiguous detection possibility for a range of microplastics (John 2000), their applicability to TWP analysis fails for three main reasons. First, extensive sample preparation is needed, usually including digestion of matrix compounds and density separation. Second, the methods encounter optical challenges from the filler components in TWP, especially carbon black, which causes near-complete absorption of IR light (Eisentraut et al. 2018; Wagner et al. 2018). Third, FTIR works best with pure substances. The existence of multiple components in environmental samples makes the method unable to detect TWP in gully pot sediments (Litvinow and De 2002). Chromatography, including pyrolysis gas chromatography–mass spectrometry (Py-GC/MS) and thermal extraction desorption (TED)-GC/MS, is presented as an alternative approach to resolve many of the problems encountered by FTIR alone (Eisentraut et al. 2018; Unice et al. 2012). Chromatography is proved to be effective in quantifying TWP polymers at a low limit of detection, and an international standard method, the ISO 21396 (ISO 2017) has been developed based on Py-GC/MS. However, the method requires deuterated internal standard procedure and establishing international market share of styrene butadiene rubber (SBR) and natural rubber (NR), which adds complexity and uncertainty. In addition, the lack of dependable marker that is specific and consistently available in all types of tires, while at the same time absent from non-contaminated (background) environmental matrices, is another problem associated with these methods. This is particularly true when the environmental matrix is suspected to develop decomposition products interfering with the chosen marker for elastomers. For

example, Unice et al. (2012) reported that the presence of monomers and dipentene in environmental samples could positively bias measured TWP concentrations. Another study, using TED-GC/MS, reported the impossibility of analyzing NR in environmental samples due to interference of dimers and higher oligomers originating from the decomposition of plant materials (Eisentraut et al. 2018).

Combining the advantages of FTIR and thermal methods has been recently reported as a good method to analyze TWP in environmental samples (Mengistu et al. 2019). The method uses first pyrolysis in simultaneous thermal analysis (STA) before FTIR scanning of pyrolysis products. The method adds a temperature dimension to create a multi-way dataset suitable to Parallel Factor Analysis (PARAFAC). PARAFAC is a powerful tool improving the application of spectroscopic methods to environmental samples and has been extensively used in food and environmental sciences (Andersen and Bro 2003; Murphy et al. 2013). It decomposes the data into underlying chemical components with estimates of spectra and concentration (Andersen and Bro 2003). Quantification is facilitated by using mass loss during pyrolysis in conjunction with scores of components from a PARAFAC model. However, no study using the method to analyze TWP in environmental samples has yet been reported.

This study addresses unanswered questions regarding TWP quantification in the environment, and its objectives are to (1) evaluate whether gully pots retain TWP from road runoff, by using a novel method combining STA, FTIR and PARAFAC, and (2) determine TWP concentrations in gully pot sediments.

2. Materials and methods

2.1. Study area

TWP concentrations in gully pot sediments were studied at 5 sites (4 municipal streets and a parking lot) in a municipality of Southern Norway (Ås, N59.660, E10.784, Fig. 1). Each site was represented by two gully pots. The characteristics of the selected sites are described in Table 1. All the selected streets had a speed limit of 30 km/h, which is common in built-up areas and town centers in Norway. Traffic density in these streets was recorded using a radar from Amparo solutions, during June and July 2020. Information on gully pot maintenance was not available at the time of the study.

2.2. Sampling

500 mL of wet sediment was collected from each gully pot (10 in total, from 5 study sites), using a spade. The samples were consistently taken from the top of the sediment accumulated under the standing water, in an attempt to account for the most recent contaminations. All samples were dried at 105 °C, crushed and homogenized with mortar and pestle, and sieved at 5 mm to exclude materials beyond the range of defined microplastics. From each sample, three replicates of 80 mg dry sediment were placed in aluminum crucibles for STA-FTIR analysis. A standard natural soil, LUFA 2.2 (LUFA Speyer, Germany) was included in the experimental set-up as a rubber-free control, also in triplicates of 80 mg dry mass. The total number of samples analyzed by STA-FTIR was 33.

2.3. STA-FTIR analysis

The acquisition of thermal and FTIR spectra involved hyphenated instruments, a simultaneous thermal analyzer STA 449 F1 Jupiter, with carrier type S (Netzsch, Germany), coupled to a Fourier transform infrared spectrometer Bruker Tensor 27 with external gas cell (Bruker, USA), as described below.

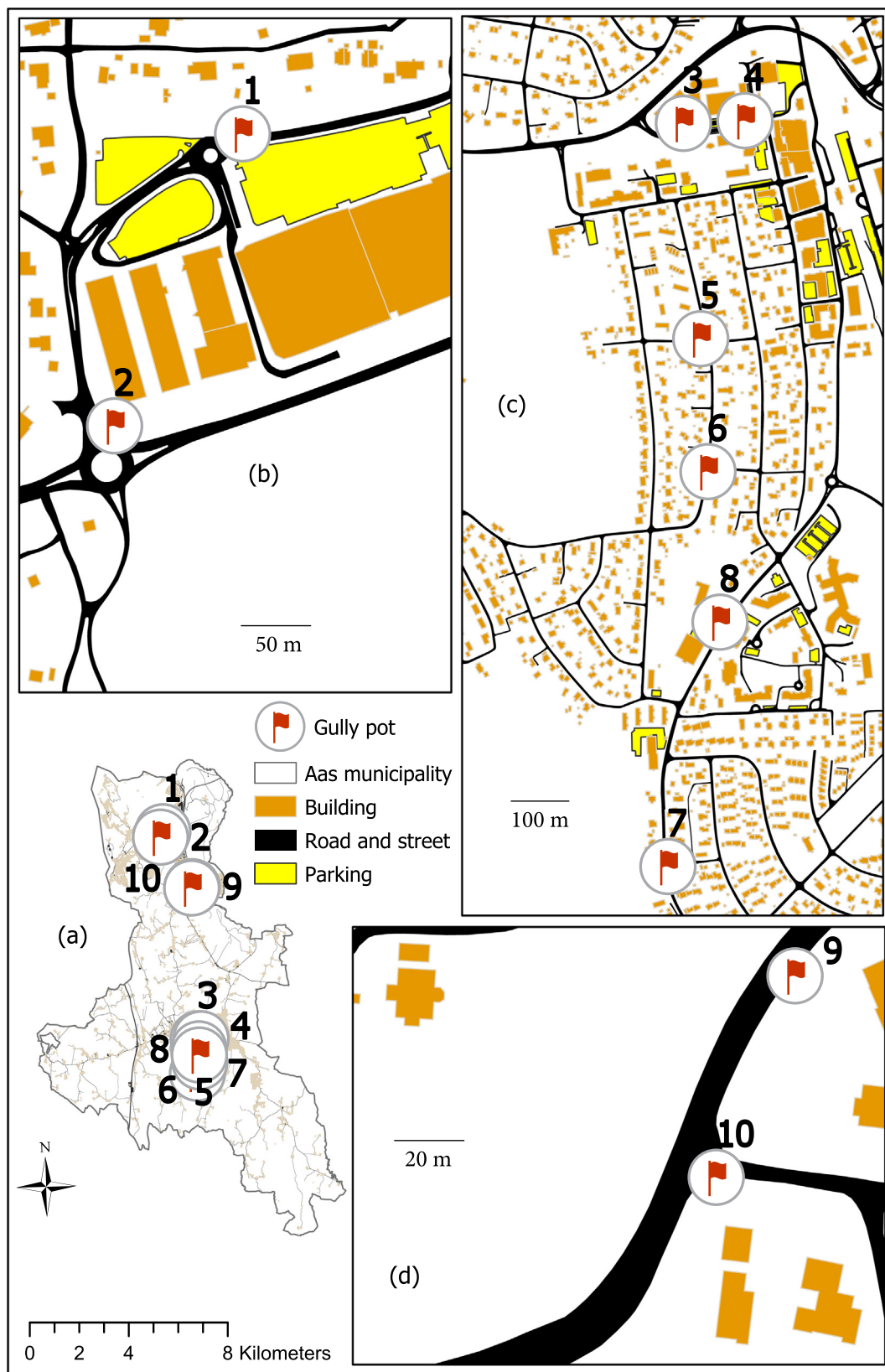


Fig. 1. (a) Overview of sampling sites and gully pots, (b) gully pots at site Sjoskogenveien (1,2), (c) gully pots at sites Raadhusplassen (3, 4), Maalrostveien (5, 6), and Brekkeveien (7, 8), (d) gully pots at site Nordbyveien (9, 10).

2.3.1. STA

Samples were heated from 100 to 800 °C at a rate of 10 °C/min under protective nitrogen atmosphere (flow rate 20 mL/min) for 70 min.

Evolved gases were purged continuously to the FTIR, using nitrogen as a carrier gas (at a flow rate of 50 mL/min). The thermogravimetric unit in the STA registered changes in sample mass (balance resolution

Table 1
Characteristics of sampling sites, gully pot ID and annual average daily traffic (AADT).

Site	Gully pot ID	AADT	Description
Sjoskogenveien	1	2608	Two roundabouts by the largest shopping center in the municipality, with 79 shops and restaurants open 6 days a week (Fig. 1b)
	2		
Raadhuplassen	3	N/A	Parking lot by the city hall, surrounded by shops and cafés (Fig. 1c)
	4		
Maahtrostveien	5	417	Street in a residential area with private homes only and little traffic (Fig. 1c)
	6		
Brekkeveien	7	1500	Street in a residential area with elderly care center and private homes (Fig. 1c)
	8		
Nordbyveien	9	890	Street in an industrial and residential area (Fig. 1d)
	10		
Control	11	N/A	LUFA 2.2 standard natural soil (LUFA Speyer, Germany), used as rubber-free control sample

25 ng) every 6.83 s during the heating process, resulting in 614 mass measurements per sample, each measurement being associated to a temperature.

2.3.2. FTIR

The average signal of 8 scans of FTIR spectra in the wavenumber range of 4000–600 cm^{-1} was collected with a resolution of 1.93 cm^{-1} during the heating process, generating 1762 signal points. Each scanning took 6.83 s, which resulted in 614 spectra per sample.

2.4. PARAFAC model

Temperature was assigned to each spectrum using the time relationship between gas release and scanning, giving the FTIR dataset Wavenumber (cm^{-1}) and Temperature ($^{\circ}\text{C}$) axes with dimensions of 1762×614 values. The data from all 33 samples were then arranged as a trilinear multi-way dataset of 33 samples \times 1762 wavenumbers \times 614 temperatures, suitable for PARAFAC decomposition (Baum et al. 2016; Mengistu et al. 2019). PARAFAC models were built using 2 to 7 components, using MATLAB (The MathWorks, Inc., R2018a, Natick, USA) and a PLS Toolbox for MATLAB version 8.6 (Eigenvector Research, Inc., Wenatchee, USA). The various steps involved in PARAFAC analysis of the STA-FTIR data are detailed in S1, while the equation of the model and explanation of scores and loadings are presented in S2. The validity of the PARAFAC model was evaluated using figures of merit (core consistency, model fit and residuals) and split-half analysis, as detailed in S3. Finally, the model with the most components and the best figures of merit was selected. The outcome of PARAFAC was used for the identification and quantification of components in the samples, and more specifically to determine if rubber materials were present in the sediments, and if so, at which concentrations. PARAFAC was also applied to a spike recovery analysis, as shown in S4.

2.5. TWP identification

Rubber materials (RM), which are the core components in tires (Thorpe and Harrison 2008), are good markers to determine the presence and estimate the quantity of TWP in sediments and soils (Wagner et al. 2018). Loadings of the PARAFAC model carry information about physical and chemical properties of the substances under evaluation. Wavenumber (cm^{-1}) and temperature ($^{\circ}\text{C}$) loadings of components were evaluated against established spectra of rubber constituents in tires, e.g. styrene butadiene rubber (SBR), natural rubber (NR) and ethylene propylene diene monomer rubber (EPDM), reported in different studies (Fernández-Berridi et al. 2006; Gunasekaran et al. 2007; Januszewicz et al. 2017; Jasminská et al. 2013; Litvinow and De 2002; Mengistu et al. 2019).

2.6. Choice of method for TWP quantification

After scores were obtained from the PARAFAC model, two alternatives were available to estimate the quantity of RM, and subsequently TWP. The first alternative was to establish a score-concentration relationship for at least one known sample, and use this relationship to calculate concentrations in unknown samples (Mengistu et al. 2019; Murphy et al. 2013). A preliminary test based on this approach, using crumb rubber, showed poor results, especially for small sample size of <1 mg crumb rubber. This might be attributed to loss of sensitivity due to small sample mass, which is common in thermal analysis (Menczel, 2009). Non-linearity between FTIR absorbance and concentration above 1% gas concentration (Schindler et al. 2013) meant that the alternative was not applicable at higher concentration either, and was thus rejected. The second alternative was based on sample mass loss during pyrolysis in STA, and the additivity and proportionality assumptions in PARAFAC (see S2, for more details). This alternative was found to be the most appropriate method to estimate the concentration of RM in gully pot sediments, and also presented the advantage of not requiring calibration with a known concentration of RM. Once a component was identified as being RM, the score of this component was used to calculate the concentration of RM in gully pot sediments, using Eq. (1).

$$C_{\text{RM}} = (\text{ml} \times a_{if}) / (m \times S_i) \quad (1)$$

where

C_{RM} = concentration of RM in a sample (mg/g)

ml = sample mass loss during pyrolysis (mg)

m = initial sample dry mass (g)

a_{if} = score of component identified as being RM in sample i

S_i = sum of scores of all components of sample i

RM constitute only a fraction of TWP, the rest being fillers, reinforcement materials, plasticizers, vulcanization and antiaging chemicals (Fernández-Berridi et al. 2006; Kreider et al. 2010; Jusli et al. 2014). While the RM fraction in TWP is variable (40–60%), 58% was used here, based on the most relevant data for the context of the study (Ragn-sells, 2020, tire crumb producer). Differences in TWP concentrations among gully pots were tested using one-way analysis of variance (ANOVA), followed by a Tukey pairwise comparison test to identify differing gully pots. The assumptions of normality and homoscedasticity were verified by normal probability plot and Levene's test, respectively, and fulfilled. The level of significance was set to 0.05.

3. Results and discussion

3.1. PARAFAC model

After testing PARAFAC models with 2 to 7 components, a model with 2 components was found to be the best to decompose the dataset, and the use of additional components did not improve the model. The model with 2 components captured 91% of the total variance in the dataset and showed good figures of merit (S3). The model showed 100% core consistency (S3a), very little overlap between model fit and unique fit (S3b), and low residuals sum of squares (S3c), indicating that the model was very stable. Split-half analysis of the dataset after running the model showed 93% similarity between the sub-datasets (S3d, e). This level of similarity confirms the validity of the model (Bro and Kiers 2003; Murphy et al. 2013).

3.2. Presence of TWP in gully pot sediments

The presence of RM in gully pot samples was revealed after concurrent assessment of the two loadings of the PARAFAC model. As presented in Fig. 2a, component 1 showed strong peaks at 2925 and 2860 cm^{-1} , and a weak peak at 3015 cm^{-1} . Even though the fingerprint

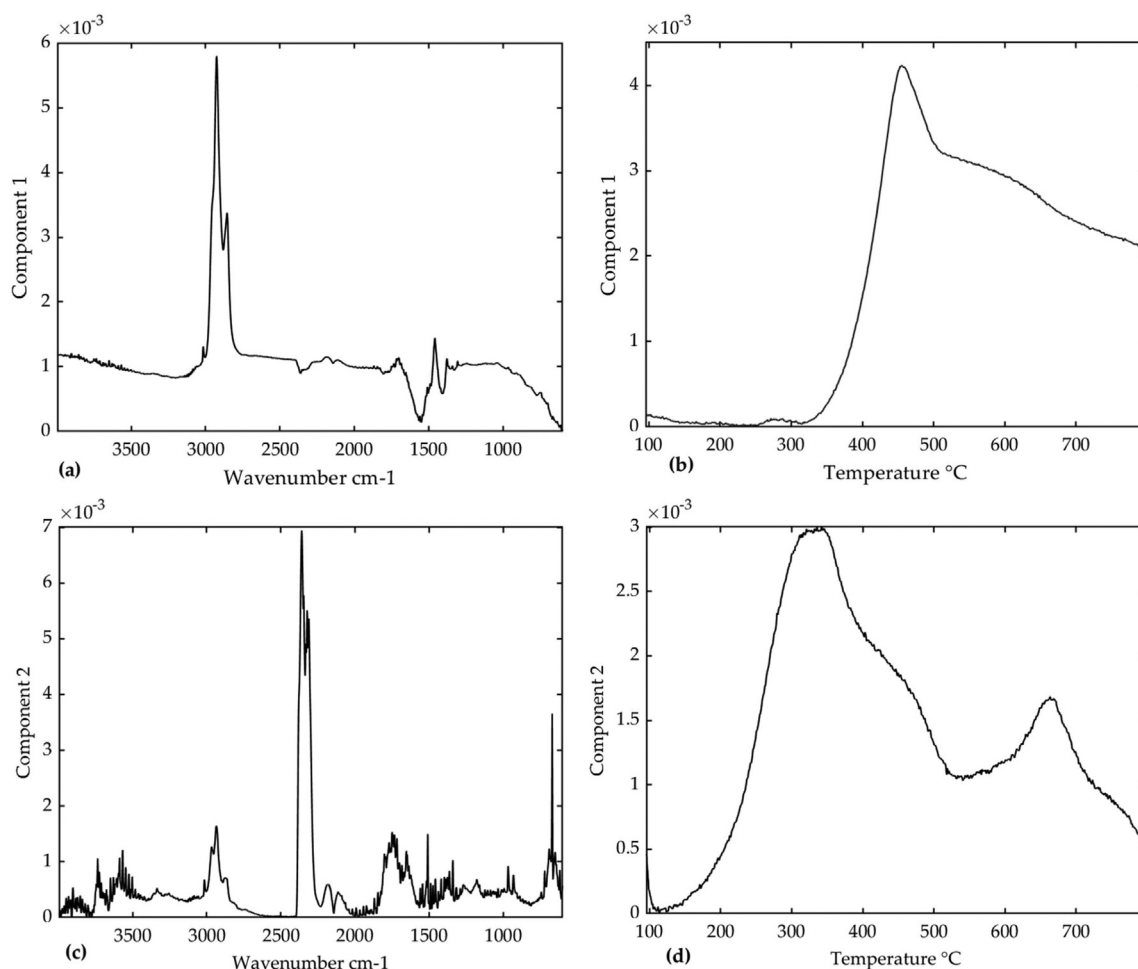


Fig. 2. Components loadings from the PARAFAC model. (a) wavenumber loading of component 1, (b) pyrolysis temperature loading of component 1, (c) wavenumber loading of component 2, (d) pyrolysis temperature loading of component 2. Component 1 corresponds to tire rubber materials and component 2 to other substances (including plant materials).

region ($<1500\text{ cm}^{-1}$) had very few clear peaks, component 1 showed weak peaks at 1450 and 1370 cm^{-1} . This spectrum was similar to those of RM identified in a study where tire crumb and tire crumb in formulated sediments were used to evaluate the applicability of PARAFAC in TWP detection and quantification (Mengistu et al. 2019). Other studies also attributed similar FTIR absorbance spectra to styrene butadiene rubber (SBR), natural rubber (NR) or ethylene propylene diene monomer rubber (EPDM) (Fernández-Berridi et al. 2006; Gunasekaran et al. 2007; Litvinow and De 2002). The thermal features shown in Fig. 2b, indicated that component 1 was a degradation product of substances pyrolyzed at temperatures starting from around 330 °C and reaching a maximum at 470 °C . In a coupled STA-FTIR, thermal spectra may show a temperature shift up to 10 °C due to transfer delay (Schindler et al. 2013), indicating that pyrolysis temperatures shown here were slightly higher than would be observed in STA alone. Compared to pyrolysis temperatures reported in literature, the thermal features of component 1 were found to be typical for pyrolysis of tires (Fernández-Berridi et al. 2006; Januszewicz et al. 2017; Jasminská et al. 2013; Jusli et al. 2014). In addition to positive matching, exclusion of components which were not rubber materials was important for the identification.

FTIR features of component 2 (Fig. 2c) were very distinct from those of component 1. Strong peaks appeared at 2360 and 670 cm^{-1} , while weaker peaks were observed at $3700\text{--}3500$, $3000\text{--}2800$, 1750 , and $1500\text{--}1300\text{ cm}^{-1}$. The strong peaks on component 2 were attributable to CO_2 (NIST, 2011; Schindler et al. 2013; Wang and Lou 2016), while the weaker peaks at 3016 , 2950 and 2870 cm^{-1} could represent a functional group of saturated and unsaturated hydrocarbons (Fernández-Berridi et al. 2006; Gunasekaran et al. 2007; Litvinow and De 2002;

Schindler et al. 2013). The thermal features shown in Fig. 2d, indicated that component 2 was a product of pyrolysis at temperatures starting from 200 °C but occurring mainly at 320 , 345 and 660 °C . These pyrolysis temperatures were attributable to plant organic matter and carbon black (Cambridge Polymer Group 2014; Jusli et al. 2014; Kawamoto 2017; Pantea et al. 2003; Yang et al. 2018). Therefore, both wavenumber and temperature loadings of component 2 suggested that this component was not RM.

PARAFAC is expected to decompose FTIR data from a multi-substance sample, like gully pot sediments, into multiple components. Contrarily, decomposition of the data in this study resulted in two components only, suggesting aggregation of components based on related features, the pyrolysis temperature in this case. This suggestion was strengthened by the fact that the rubber-free control samples (LUFAs soil) only had one component in this model, while these samples had two components when modeled alone (CO_2 and H_2O , results not shown). The non-specificity of IR and its quality in identifying functional groups (Litvinow and De 2002) has probably contributed to the observed aggregation. This aggregation phenomenon is against the basic definition of PARAFAC modelling and prevents identification of individual polymer types (SBR, NR, EPDM) in TWP. However, given that TWP in gully pots originate from different types of tires (summer and winter tires, heavy duty vehicles and passenger cars tires) whose polymer composition is under commercial confidentiality, aggregation was here a convenient attribute, presenting all RM as one component. The aggregation may also have helped detect polymers present in smaller quantities, which may otherwise be undetectable.

The presence of similar dimers in both TWP and plant materials has recently been reported as a challenge to establish dependable markers (Eisentraut et al. 2018). Concurrent evaluation of the two loadings was therefore critical for TWP identification, as no single spectrum was robust enough to unambiguously represent the substance under investigation. For example, hydrocarbon functional groups were not only visible on wavenumber loadings of component 1 (RM), but also of component 2. This phenomenon was resolved by analyzing temperature loadings showing that the hydrocarbons in the two components were pyrolysis products of different substances. Thermal spectra (Fig. 2b and d) indicated that component 1 (RM) resulted from maximum degradation at 470 °C, while component 2 resulted from degradation of plant materials and carbon black at 330 and 660 °C, respectively.

3.3. TWP concentrations in gully pot sediments

Individual sample mass loss during pyrolysis ($7.1\% \pm 3.3\%$, mean \pm standard deviation (SD), $n = 33$) was used together with PARAFAC scores to calculate concentrations of RM, and in turn, TWP, in all sediment and control samples. The spike recovery analysis indicated a high but acceptable recovery rate of 123% (Supporting Information, S4). RM and TWP concentrations in sediments (in mg per g sediment dry weight) are presented in Table 2 and were found to differ significantly across gully pots ($F_{10,32} = 194.6$, $P < 0.001$). TWP concentration in the control soil was 0.2 ± 0.3 mg/g, which based on US EPA procedure for the determination of the method detection limit, MDL (EPA, 2016) indicates a MDL of 1.1 mg TWP per g dry sediment. Although very high compared to e.g. py-GC/MS, the detection limit is very good for a thermal-spectroscopic method with no pre-processing. The highest TWP concentration, 150 mg/g, was found in gully pot 1, in Sjoskogenveien, followed by 131 mg/g in gully pot 4 at Raadhusplassen. Gully pots 2 (Sjoskogenveien) and 3 (Raadhusplassen), with 71 and 42 mg/g, respectively, also contained significant amounts of TWP, while concentrations in gully pots 5–10 ranged from 1 to 20 mg/g, and were not found statistically different from each other and from the rubber-free control soil. The concentrations found here are in the range of those reported in roadside ditch soils, namely 0.3–117 g/kg (Vogelsang et al. 2018) and 11 g/kg in Swedish road detention systems (Wik et al. 2008). Concentrations of this magnitude are however in contrast with older studies suggesting poor TWP retention in gully pots (Cadle and Williams 1978; Fauser 1999).

The highest TWP concentration was found in the street with the highest AADT (2608, Sjoskogenveien), suggesting the influence of traffic density on TWP concentrations in gully pots. This is consistent with the results of Bondelind et al. (2020) in which higher microplastics concentrations were associated with higher traffic density. But, it was evident that AADT was not the only factor influencing TWP concentrations,

Table 2
Concentrations of rubber materials (RM) and tire wear particles (TWP) concentrations in gully pot sediments (mg/g sediment dry weight). Results are provided as mean and standard deviation (SD), $n = 3$ per gully pot and control. Means with different letters are statistically different (Tukey, $P < 0.05$).

Site	Gully pot ID	RM (mg/g sediment dw)		TWP (mg/g sediment dw)			
		Mean	SD	Mean	SD		
Sjoskogenveien	1	86.7	a	5.3	149.6	a	9.1
	2	41.4	b	1.3	71.4	b	2.2
Raadhusplassen	3	24.4	c	2.3	42.0	c	4.0
	4	75.8	a	9.9	130.7	a	17.0
Maaltröstveien	5	0.8	d	1.4	1.4	d	2.4
	6	1.6	d	2.8	2.8	d	4.8
Brekkeveien	7	0.5	d	0.5	0.8	d	0.9
	8	5.8	d	5.0	10.0	d	8.7
Nordbyveien	9	11.5	d	0.5	19.8	d	0.9
	10	1.5	d	1.5	2.7	d	2.5
Control	LUFA soil	0.1	d	0.2	0.2	d	0.3

and that driving patterns, such as braking and acceleration intensity and frequency were also contributing factors. The high TWP concentrations in gully pots at Sjoskogenveien (roundabouts) and Raadhusplassen (parking plot), where high braking activities are expected, supported this suggestion. This finding was in agreement with Knight et al. (2020), who reported an association between high level of braking and acceleration, and generation of high amounts of TWP. In addition, TWP concentrations were significantly different in the 2 gully pots of a same site at Sjoskogenveien (by a factor 2) and Raadhusplassen (by a factor 3) indicating possible effect of micro-environment on mobilization of TWP entering the gully pots. However, the influence of other factors, like management activities, could not be excluded, as not specifically evaluated here. These factors, e.g. emptying frequency, particle size and density, stormwater inflow intensity and design of the gully pots have been reported to influence particle accumulation in gully pots (Bolognesi et al. 2008; Lindholm 2015).

3.4. Implication for environmental pollution management

Gully pots are established to retain solid particles in order to facilitate the well-functioning of drainage systems and protect downstream waters against pollution. They have been found to retain a range of environmental pollutants (Lindholm 2015) and are often the only pollution control infrastructure before road runoff reaches downstream recipient water. This study demonstrated that TWP were present among the solid particles retained in gully pots, supporting the finding by (Kreider et al. 2010; Sommer et al. 2018; Wagner et al. 2018) that sedimentation may occur following heteroaggregation of TWP with higher density particles in the road environment. The presence of TWP at concentrations reaching 150 mg/g, i.e. 15%, of the sediment indicated that gully pots could serve as temporary sinks to protect recipient water from pollution, yet posing an acute pollution risk if not timely emptied. The present study was conducted in streets with low traffic densities and low speed limits, but the approach would be highly relevant to road environments with more intense traffic conditions, such as highways and urban roads. Eventually, knowledge of TWP concentrations and their evolution over time would help road managers design timely and efficient emptying routines and safe disposal plans for gully pot sediment. Furthermore, the method could be applied for generating data for environmental fate and mass balance models, which would necessarily need to account for TWP trapping efficiency in stormwater.

4. Conclusion

By combining STA, FTIR and PARAFAC, rubber materials signals were identified and TWP concentrations were determined in gully pot sediments, without prior sample preparation. The model appeared to perform well, spike analysis showed good recovery rate, and the method provided a feasible way to apply FTIR to TWP. Concentrations of TWP in the studied gully pot sediments ranged from below 0.1% to 15%, showing the potential of gully pots to act as temporary sinks for TWP, while also posing an acute pollution risk for downstream water recipients, if not properly managed. The highest TWP concentrations were found at sites with higher traffic density and braking/acceleration intensity. However, to conclusively determine the influence of traffic conditions on TWP concentrations in gully pots, the approach should be applied in settings where gully pot emptying frequency and all TWP generating and mobilization factors are controlled. For sediments with low TWP concentrations (<1 mg/g), density separation might be an interesting pre-step to concentrate TWP in samples, and could also help to clarify the amount of TWP in different density classes. Another interesting pre-step might be mechanical sieving of the samples, which could also be helpful to understand capture efficiency by size in gully pots. Improving laboratory equipment and analytical procedures is another area to consider, where the use of high resolution STA, for instance, might

help individual polymer detection by improving separation between closely occurring mass loss events. Finally, by enabling quantification of TWP in gully pot sediments, the approach supports environmental monitoring of TWP and safe disposal of gully pot sediments, which is critical for environmental pollution management.

CRediT authorship contribution statement

Demmelash Mengistu: Conceptualization, Formal analysis, Methodology, Writing – original draft. **Arve Heistad:** Funding acquisition, Supervision. **Claire Coutris:** Conceptualization, Funding acquisition, Writing – review & editing.

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.

Acknowledgements

The authors wish to thank Monica Fongen for the STA-FTIR analysis of the samples.

Funding

The Norwegian Institute of Bioeconomy Research (NIBIO) financed the STA-FTIR analysis, through funding from the Norwegian Research Council (Grant number 194051). Financial support from the municipality of Ås, Norway, and the Norwegian Research Council (Grant number 272946) is also acknowledged.

Appendix A. Supporting information

Supporting information to this article can be found online at <https://doi.org/10.1016/j.scitotenv.2020.144785>.

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