Determination of fats, oils and greases in food service establishment wastewater using a modification of the Gerber method

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Abstract

Discharges from food service establishments (FSEs) are a major source of fat, oil and grease (FOG) which cause blockages in sewer networks. Previous research has identified that current methods are unsuitable for quantifying FOG in FSE wastewater owing to interference from surfactants in detergents, and protein from food residuals which emulsify FOG. A novel quantification method, based on the dairy industry Gerber method, has been developed which negates the impact of surfactants. Moreover, the method allows free and emulsified oil to be quantified separately providing greater insight into FOG management strategies. Trials in synthetic and real FSE wastewaters indicate the novel method is more reliable than standard liquid–liquid and solid phase extraction in FOG-rich systems.

Introduction

The term FOG (fat, oil and grease) encompasses a number of different materials (liquid and solid) and describes a heterogeneous group of chemicals including tri-, di- and mono-glycerides, sterols, non-volatile hydrocarbons, waxes and other complex lipids which exist in a combination of free and emulsified forms. Suspended and emulsified FOGs [food service establishment (FSE) wastewater] are discharged into sewer systems from both domestic and commercial premises and are attributed to cause a significant proportion of the deposits (solidified and saponified) forming sewer blockages (Williams *et al.*, 1995). Discharges from FSEs are known to represent a major source of FOG deposits in sewers and hence area focus for managing their discharges. Current management practices of FSE wastewater include treatment in the form of biological additives to process the FOG material into benign end products; or passive separators to collect it. Current challenges associated with the cost and complexity of suspended and emulsified FOG measurement mean that surrogates are often preferred when selecting, designing and confirming performance. For instance, passive gravity separators commonly use diesel oil as a surrogate in validation tests although the specific gravity of the oil is significantly lower than typical FOG, raising questions over its applicability (Barton, 2012).

FOGs are insoluble in water but soluble in solvents (e.g. chlorinated fluorocarbons and alcohols) such that current measurement methods incorporate a solvent extraction step (BS EN 1825-1:2004, 2004; US EPA/R-98-002, 1999). In the United Kingdom, the standard method involves a liquid–liquid extraction (LLE) with 1.1.2-trichloro-1.2.2-trifluoroethane ($C_2Cl_3F_3$) which, due to environmental concerns, has been replaced with more appropriate options such as carbon tetrachloride (CCl_4) or n-hexane (C_6H_{14}). As each solvent potentially extracts different materials it is common practice to refer to the solvent used when expressing a concentration, for example, hexane extractable material (HEM). Quantification of the FOG content in FSEs wastewater is normally based on gravimetric measurement of residuals post-solvent evaporation, with reported alternatives utilising infrared spectroscopy or gas chromatography.

The current solvents exacerbate an issue with incomplete phase separation, requiring remedial actions if an emulsion persists such as increased agitation (Ducoste *et al.*, 2008) or solid phase extraction (SPE) (Barton, 2012). The latter involves use of a hydrophobic matrix that retains all non-aqueous components as the sample passes through the material. The retained oils are back eluted with an appropriate solvent (i.e. n-hexane) and the post-evaporated residual weighed (Wells *et al.*, 2013). A benefit of such approaches is the ability to pre-concentrate enabling more accurate measurement of low concentrations of FOG.

Previous investigations concerning current FOG levels and their treatment have indicated that predicted oil removal does not match measured oil removal

Table 1 Reported HEM recovery with different methods and interfering substances

Method	HEM recovery (%)	Interfering component	Reference
EPA 1664A (LLE-Hexane)	44–58	Protein	Wang and Ducoste (2013)
Modified LLE	111–117	Protein	Wang and Ducoste (2013)
Modified LLE method	47–63	Surfactants	Barton (2012)
SPE	91–92	None	Barton (2012)
SPE	30–50	Surfactants	Barton (2012)
SPE	63–78	Protein	Barton (2012)
Modified Gerber	92–104	Protein and surfactants	Davis et al. (2011)

(Lopez-Vazquez and Fall, 2004; Ducoste et al., 2008). Investigation into FOG recovery efficiency has demonstrated measurement interference when protein and/or detergent surfactant are present (Table 1). The surfactant molecules form micelles around the FOG droplets inhibiting hexane solvation; and proteins can be carried into the solvent increasing the total mass transferred. Furthermore, emulsion separation can be inhibited through foam formation during the agitation phase which may be ameliorated through centrifugation (Barton, 2012). Previous reports have suggested that SPE is more effective than LLE in the presence of surfactants (Lau and Strenstrom, 1997), although more recent studies have demonstrated that recovery remains low with SPE in such cases (Barton, 2012). For instance, current investigations have concluded that surfactant concentrations of sodium dodecyl sulphate (SDS) beyond 40 mg/L inhibit recovery, with recovery levels as low as 20% once the SDS concentration exceeds 400 mg/L. Accordingly, FOG measurements can both under and overestimate the true content thereby influencing the understanding of true loads and the efficacy of the different treatment options. To illustrate, an overestimation of feed coupled to an underestimation of the effluent could considerably overestimate the removal efficiency of a system.

An alternative proposition is the Gerber method, which is an established method in the dairy industry for determination of the fat contents of raw and processed milks, and is used worldwide for applications such as payment testing and process standardisation (Kleyn et al., 2001). The fat within milk exists as a stabilised emulsion due to protective protein coatings around the fat globules. Consequently, the sample is digested in sulphuric acid to break down the proteins releasing the fat, and isoamyl alcohol is added to facilitate phase separation. The contents are mixed in a specially designed butyrometer, and centrifuged to isolate the fat into the tube of the butyrometer where the percentage fat content is read from the graduated scale at a defined reading temperature of 65°C (BS ISO 2446:2008, 2008). The scale is based on a specific gravity of butterfat of 0.9 at the measuring temperature, and a predefined volume of milk (10.75–11 mL).

Establishment of the consistency of the method through an international collaborative study concluded that the relative standard deviation of the measurement was 1.8% for low fat solutions (1–2% fat) and 0.6% for high fat (2–6% fat) solution (Kleyn et al., 2001).

The Gerber method represents a simple, rapid and inexpensive approach for determining fat contents and as such has seen its use beyond just milk: with examples including cheese and meat products (de Langen, 1963); macadamia nuts (Rosenthal et al., 1985a) and avocado (Rosenthal et al., 1985b). A correction factor must be applied due to the difference in the specific gravity (s.g.) of the type of fat being measured. For instance, in the case of meat a correction factor of 0.935 was applied based on the ratio of specific gravity of lard to butterfat (de Langen, 1963). Reported recoveries exceeded 99% at fat contents above 3% such that the author concluded that the Gerber method provides a more rapid and reliable measurement than traditional solvent extraction. Similarly, positive comparison between Gerber and solvent extraction has been reported in the case of avocados with a regression coefficient for the linear trend of $r = 0.928$ (Rosenthal et al., 1985b). Utilisation of the Gerber method for these systems relies on dissolution of the fat containing material and as such modification to the procedure has been required in terms of the temperature and the agitation time prior to measurement. For instance, in the case of the meat, the sample went through four cycles of agitation followed by resting in the water bath (de Langen, 1963).

The approach has also been adopted for fat measurement in wastewater from small dairy-based FSEs (Davis et al., 2011) requiring a modification where the fat was precipitated, isolated and condensed from larger samples to enable accurate determination. The approach was to lower the system pH to the isoelectric point (i.e.p.) of casein (pH 4.6). Casein-stabilised fat globules precipitated and aggregated, and the isolated precipitate was processed through the standard Gerber method. The current paper extends such work to all FSE wastewater types by further modifications enabling the Gerber method to be applied to non-dairy-based systems. In such cases, casein is added to promote co-precipitation of the emulsified material.

Materials and methods

All reagents used were obtained from Fisher Scientific, United Kingdom, unless stated otherwise. Tests were performed on both synthetic and real FSE wastewaters. Emulsions were produced by emulsifying rapeseed oil (Tesco, Bedford, UK) at different concentrations between 10 and 750 mg/L in distilled water to a total volume of 200 mL combined with analytical grade SDS in a 250 mL glass bottle. The impact of other potential matrix interferences was assessed using a synthetic FSE WW containing soy protein acid hydrolysate (Amisoy, 400 mg/L), glucose (1200 mg/L), cornflour (Tesco, 250 mg/L), SDS (30 mg/L), Triton X-100 (14 mg/L), calcium chloride dihydrate (100 mg/L), potassium chloride (70 mg/L), magnesium sulphate heptahydrate (40 mg/L), ammonium chloride (10 mg/L), iron chloride (0.27 mg/L), zinc sulphate heptahydrate (0.16 mg/L), copper sulphate (0.07 mg/L), manganese sulphate monohydrate (0.045 mg/L), cobalt nitrate hexahydrate (0.002 mg/L) and ammonium heptamolybdate tetrahydrate (0.001 mg/L). Surfactants were measured with cell test kits (Spectroquant, Merck Millipore, Watford, UK) quantified with a NOVA 60A Spectroquant photometer.

The other reagents used were sodium caseinate, Gerber sulphuric acid (density at 20°C is 1.816 ± 0.004 g/mL), isoamyl alcohol (density ranging between 0.808 and 0.818 g/mL), 5M hydrochloric acid, n-hexane (HPLC grade), 1% sodium hydroxide. The density of rapeseed oil was determined by hydrometry per BS EN ISO 3675:1998 to be 0.889 ± 0.003 g/mL at 60°C. Real FSE wastewater was collected from local establishments on the campus of Cranfield University from a potwash sink and a commercial dishwasher.

Free oil determination

Free FOG measurement was performed separately to the emulsified phase by pre-separating the free floating phase onto a disc of pre-conditioned hydrophobic polypropylene material (Serpro Ltd, Maidstone, UK). The aqueous phase was decanted, and weighed, for processing by the Gerber method, and 40 mL of hexane added to the sampling container followed by gentle shaking to solvate FOG from both the disc and vessel surface. Following removal of the disc and phase separation, 20 mL hexane was extracted, evaporated and the residue weighed. All samples were heated to 50°C to ensure any animal fat was melted. The characteristics of the fat found in wastewater are related to the food prepared on the day. This modified Gerber method was developed using rapeseed oil, therefore the heating step is necessary when analysing unknown samples to ensure fat liquefaction. Optimisation of the method was performed in relation to elution time. Ten samples of disc material went through the procedure

to ascertain a method blank reading and the mean measured extracted material subtracted from sample measurements. Potential interference from adsorbed surfactant was determined by triplicate blanks at surfactant concentrations between 0 and 500 mg/L indicating a maximum additional mass of 2–3 mg HEM per disc when the surfactant concentration reaches 500 mg/L.

Modified Gerber method

Approximately 200 mL samples were weighed into transparent polycarbonate centrifuge bottles (250 mL Nalgene, Fisher, Loughborough, UK). The FOG concentration step of emulsified phase extraction involves mixing 1 mL of 10% sodium caseinate solution into emulsified samples and reducing the pH through addition of hydrochloric acid until precipitated particle aggregation is visually confirmed, based on a 30 s mixing period. The sample is then placed horizontally on a rotary shaker operated at 90–95 rpm for 15 min or until the aqueous phase is clear. The sample is centrifuged at 2000 g for 10 min (Sorvall Legend RT+, DJB labs, Newport Pagnell, DJB labcare, UK) which is repeated if small particles are visually present in the supernatant. The supernatant is discarded and 1% sodium hydroxide added to the precipitate and residual fluid to raise the pH to 7 to partially re-dissolve the casein generating a slurry for easy transfer.

The method then follows the Gerber method (BS ISO 2446:2008, 2008) with some modifications. The slurry is layered onto 10 mL of sulphuric acid in a 0–1% butyrometer (Funke Gerber, VWR, Radnor, USA, UK), followed by a few mL of DI water that were used to rinse down the walls of the centrifuge bottle. One millilitre of isoamyl alcohol is added and the total volume topped up with distilled water such that the liquid surface lies half-way up the butyrometer neck to facilitate setting the oil column on the graduated scale. An acid resistant bung is inserted and the butyrometer shaken vigorously for 90 s. The butyrometer is centrifuged in a heater Gerber unit (Funke Gerber Nova Safety, VWR, UK) for 10 min followed by tempering in a water bath at 65°C for 3–10 min.

An average specific gravity of 0.9 g/mL was used for all samples. The density of the rapeseed vegetable oil used for method development was measured with a hydrometer following the BS method (BS EN ISO 3675:1998, 1998) and was equal to 0.889 ± 0.003 g/mL at 60°C. Although the Gerber milk fat method specifies a reading temperature of 65°C, the value usually applied for standard measurements is the specific gravity at 60°C, given that the butyrometers inevitably cool slightly during reading.

The density of the wastewater was calculated using the paper reported in Esteban *et al.* (2012) and Noureddini *et al.* (1992) who measured densities of various edible oils at

different temperatures. The mean specific gravity of 12 oils tested at 60°C was 0.8909 ± 0.0045 g/mL. This value also agrees with the 0.9 g/mL reported by DEFRA (2002) for waste oil from oil/water separators (EWC Code 13 05 06).

The potential for surfactant interference was assessed on both free and emulsified FOG recovery by varying SDS concentrations between 0 and 500 mg/L. Recovery and detection limits were assessed by varying the initial oil concentration of the synthetic solution with rapeseed oil up to 735 mg/L.

Detection limits

The definitions of the detection limits were as expressed in USEPA guidelines (40 CFR appendix B part 136). The limit of detection (LOD or method detection limit MDL) is defined as the minimum concentration that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. This was determined by seven low-concentration replicates for emulsified oil, and statistical distribution of 10 blanks measurements for free oil. The limit of quantification LOQ (or practical quantification limit, PQL) is defined as the lowest concentration of an analyte in a sample that can be determined with acceptable precision and accuracy under the test conditions, and was ascertained through analysis of the experiments with varying initial oil concentration.

Comparison to SPE and liquid–liquid methods

Samples of both synthetic and real FSE wastewater were co-analysed by LLE and SPE to compare with established methods based on US EPA 1664. In the case of LLE, samples were adjusted to below pH 2 and transferred to a 1 L glass separating funnel. Three 10 mL portions of n-hexane were used to wash the bottle and combined in the separating funnel with a further 20 mL of n-hexane. The funnel was manually shaken for 2 min and then left to stand for 30 min. The aqueous portion was drained and the solvent phase filtered through 10 g of anhydrous sodium sulphate into a preweighed 250 mL round bottom flask. The aqueous phase went through two more solvent extractions before the combined solvent system was dried in a rotary evaporator (Hiedolph Laborota 4000 Hiedolph instruments, Schwabach, Germany). The flasks were further dried overnight at 45°C, cooled in a dessicator and weighed.

In the case of SPE, samples (adjusted to pH < 2) were passed through a SPE disc (Empore Oil and Grease, Sigma Aldrich, Gillingham, UK) in a vacuum filtration system. The discs had been rinsed with hexane, pre-conditioned with 10 mL methanol and rinsed with 30 mL DI water. The sample bottles were then rinsed progressively twice with 10 mL n-hexane which was passed through the disc

to elute retained FOG. A final 10 mL hexane was used to rinse the filtration glassware and disc. The combined hexane extract was passed through sodium sulphate into a preweighed 100 mL round bottom flask and then measured as per the liquid–liquid method. In some samples of real WW, with high loads of suspended solids, a glass wool prefilter was employed to prevent clogging of the extraction disc.

Results and discussion

Emulsified oil isolation

In the absence of surfactant, aggregation of the oil–caseinate mixture initiated at or close to the i.e.p of casein (Fig. 1). Addition of SDS reduced the i.e.p. of the mixture such that at a SDS concentration of 100 mg/L the required pH for visual aggregation varied from 2 to 3.8 (Fig. 1). At higher SDS concentrations of 200 and 500 mg/L, the highest pH that visual aggregation occurred was between 1 and 2. Typical surfactant concentrations in real FSE wastewaters vary widely; for instance, levels in the FSE samples used in the current study were 217 ± 119 mg/L from sinks and 26 ± 9 from dishwasher effluents (n = 16). The levels are below the critical micelle concentration (CMC) of SDS in both the current synthetic experiments and the real FSE wastewaters as the CMC of SDS varies between 7 and 9 mM as a function of pH (Rahman and Brown, 1983). Consequently, the observed changes in the pH required to induce precipitation reflect surfactant–casein interactions and direct inhibition through competition between casein and the surfactant for the surface of the oil (Demetriades and McClements, 2000).

Aggregation in real FSE WW samples followed a similar correlation, for example dishwasher samples contained anionic surfactant concentrations of 2 ± 1 mg/ and
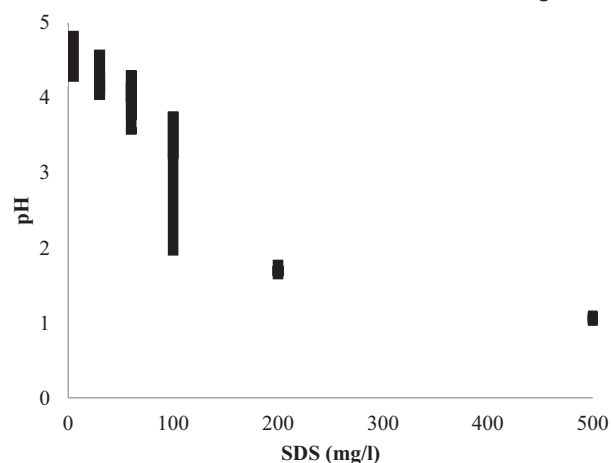
Fig. 1. Impact of SDS concentration on the required pH range to induce aggregation of a synthetic oil–caseinate emulsion.

consistently aggregated over a pH range between 4 and 4.4. Sink samples had higher, and more variable levels: aggregation was achieved between pH 3.3 and 3.8 for samples up to 183 mg/L, but adjustment to 1.7 was required for a sample measuring 278 ± 18 mg/L. The pH change is required for the oil replacement in the surfactant–casein interaction. The amount of hydrochloric acid required to precipitate the oil–casein particles varies in all samples and is visually confirmed once precipitation starts, based on a 30 s mixing period.

Free oil method optimisation

Determination of the most appropriate approach to using the adsorbent material for free oil recovery was ascertained in relation to disc preparation, mixing intensity, contact time, and the impact of surfactants. 'No oil' blank experiments revealed background measurements of fine particulate material up to 21 mgHEM/disc.
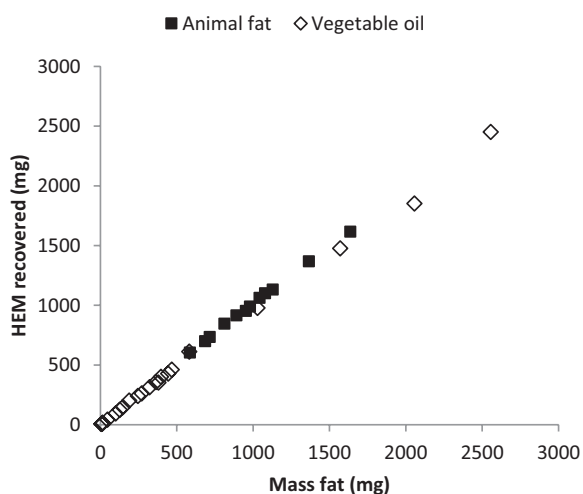

Fig. 2. Free FOG recovery from adsorbent discs.

Pre-conditioning, by a five-minute soak in hexane and vacuum drying, reduced background HEM to 14.3 ± 1.3 mg/disc ($n = 6$) which was further reduced to 7.7 ± 2.5 mg/disc and 3.8 ± 2.1 mg/disc ($n = 10$) by reduced shaking and additional washing, respectively. Validation against known masses of approximately 450 mg free oil in DI water revealed no significant difference in recoveries (100 ± 3%) for disc elution times between 5 and 30 min. The impact of surfactants on free oil recovery was elucidated by conducting a series of trials at different SDS concentrations. The measured HEM increased from 8.0 ± 2.0 mgHEM/disc without surfactant to a maximum of 11.5 ± 1.5 mgHEM/disc in the presence of 500 mg/L of SDS. At, and below, 200 mg/L no statistically significant difference was observed compared to the surfactant-free samples.

Trials of free FOG recovery revealed recovery rates of 101 ± 1.5% for animal fat and 97 ± 6.4% for oil (Fig. 2). The maximum added animal fat level was 1637 mg/200 mL sample, equating to a concentration in excess of 8000 mg/L indicating that the approach is suitable for FSE and wastewater samples. Indeed, it is posited that the use of a separate free oil measurement provides much greater insight into the load, treatment and impact of FOG in FSE wastewater and downstream into the sewer than using traditional measurements which do not differentiate between the two phases.

Emulsified FOG recovery was 95 ± 4.2% over a range of concentrations from 70 to 750 mg/L, below which recovery decreased with a minimum detectable concentration around 10 mg/L (Fig. 3). Measurement from diluted stock emulsion replicated revealed a limit of detection of 20 mg/L at a recovery rate of 27% in all seven replicated tests. The associated limit of quantification was determined to be 60 mg/L based on acceptable accuracy and precision of recovery at 80 ± 3.9% ($n = 7$). However, the use of the precipitation stage enables a threefold
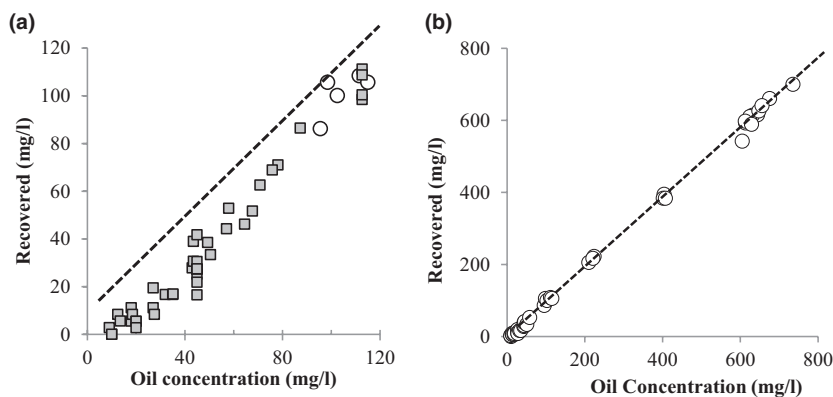

Fig. 3. FOG recovery as a function of FOG concentration in the synthetic wastewater. (A) Open black circles represent initial run between 100 and 750 mg/L and (B) grey boxes represent a second run between 10 and 110 mg/L.

pre-concentration without a loss of accuracy such that the working LOQ is estimated at 20 mg/L. This compares to LOD and LOQ for LLE of 1.4 and 5 mg/L based on surfactant free trials of 1 L samples (US EPA/R-98-002, 1999) with an acceptable recovery range of 78–117%. However, previous trials have demonstrated the reduced recovery of LLE in the presence of surfactant – detergent and protein – (Barton, 2012; Wang and Ducoste, 2013) suggesting a balance between LOQ and recovery is required. Analysis for the free oil measurements indicated accuracy and precision of 97 ± 6.4% and estimated LOD of 36 mg/L from statistical analyses of method blanks. As recovery remained within a range of 80–107% down to 36 mg/L, the LOQ can be assumed equivalent to LOD.

Dual method validation

The use of the Gerber method for emulsified oil coupled to a separate measurement of free oil through pre-adsorption enabled FOG recovery levels of 101 ± 2% across all surfactant concentrations tested at a total initial oil concentration of approximately 2000 mg/L (Fig. 3A) and 100 ± 7.4% at 500 mg/L (Fig. 3B). Comparison to previous trials reveals far better recovery than with LLE or SPE in the presence of surfactants where recovery levels were 47–63 and 30–50%, respectively (Barton, 2012). The trial in Fig. 4A demonstrated that the majority of the oil remained as free oil during manual agitation with a maximum emulsified fraction of 16% observed in the absence of surfactant. To ensure a greater fraction of emulsified oil a subsequent trial was conducted where samples was prepared by stirring for 60 min on a magnetic plate with a lower oil concentration of 500 mg/L. In addition, pH was adjusted to <2 to reflect typical environmental preservation procedures for sampling (Fig. 4B). Much greater levels of emulsified oil were observed in the samples, which equated to between 52 and 93% of the recovered oil when the surfactant concentration was 200 mg/L or less. The SDS concentration used in the experiment was higher than the range identified by our sampling campaign (20–250 mg/L) and similar to values reported in other studies (Lee et al., 2013). The concentration of oil used in this set of experiments was higher than that of real FSEs for a better understanding on the extraction ability of the proposed method between emulsified and non-emulsified oil. Overall recovery was less consistent, but remained between 96 ± 4% to 107 ± 5% at surfactant concentrations ≤200 mg/L and 88 ± 4% at 500 mg/L (Fig. 4B). Such recovery levels are consistent with trials on a dairy-based FSE using the Gerber method alone which reported recovery levels of 92–104% (Davis et al., 2011). Comparison to trials on low fat milk (1–2%) reveals a relative standard deviation across lab trials of 1.8% (Kleyn et al., 2001) suggesting that modification of the method to enable use across FSE has not adversely impacted on the recovery appreciably.

Comparison of methods

Comparison of the Gerber method and LLE on a series of synthetic FSE samples with varying initial oil concentration revealed significantly poorer recovery in the case of LLE (Fig. 5). For instance, at initial oil concentrations of approximately 10 and 30 mg/L recovery levels were 279 and 477% for the LLE and 51 and 59% for the Gerber method without pre-concentration. In contrast to the overestimation at low oil concentrations, the LLE method tended to under estimate oil levels for samples at initial oil concentration of between 50 and 400 mg/L. In these cases, the LLE recovery varied between 49 and 74% whereas the recovery level for the Gerber method was 85% at 50 mg/L and then between 96 and 99% thereafter.

At oil concentration of 30 and 10 mg/L, the SPE extraction showed better recovery levels than the Gerber method: 99% vs. 49% and 146% vs. 51%, respectively. This
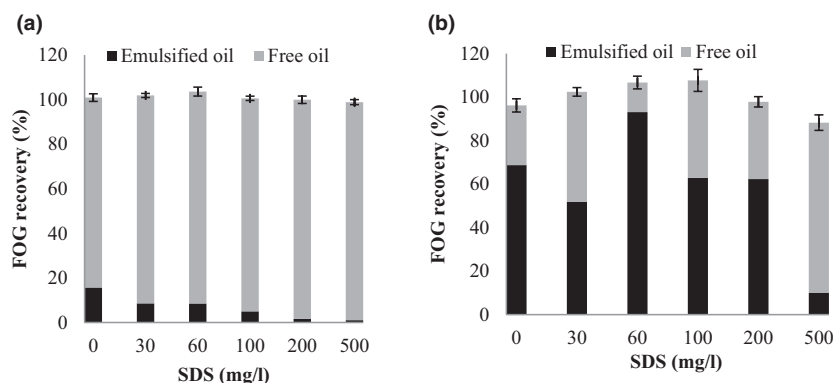

Fig. 4. FOG recovery as a function of SDS in (A) manually mixed (oil concentration 2000 mg/L) and (B) mechanically mixed systems (oil concentration 500 mg/L).
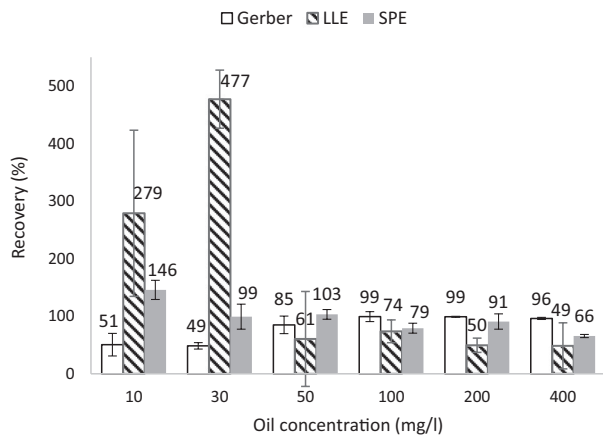
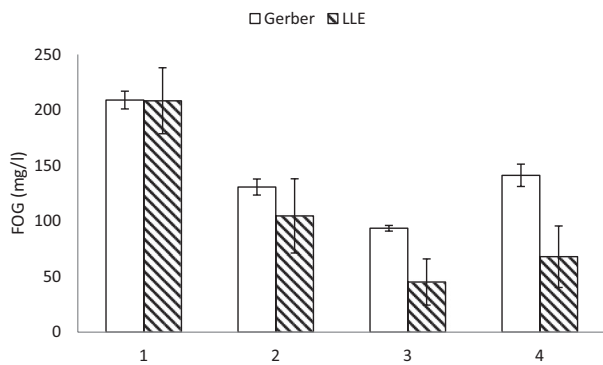**Fig. 5.** Comparison of the Gerber method, LLE and SPE for synthetic FSE samples (*n* = 3).

Fig. 6. Comparison of the Gerber method and LLE for real FSE wastewater (*n* = 3).

indicates that the LOQs for the two methods are 50 mg/L for the Gerber and 30 mg/L for the SPE one. Overall, at concentrations higher than 100 mg/L the Gerber extraction showed smaller errors than the SPE method. Overall, SPE proved more reliable than LLE at all oil concentrations.

Comparison to real FSE wastewater reveals less difference between the two methods although overall the LLE still underestimated the values compared to the Gerber method and showed greater variation within the triplicate measures (Fig. 6). To illustrate, the fourth sampling of the FSE wastewater generated the greatest difference with the Gerber method recording a FOG level of 141 $\pm$ 11 mg/L compared to 68 $\pm$ 29 mg/L with the LLE.

Analysis of the combined Gerber and free oil method in comparison to SPE for real FSE sink and dishwasher effluents revealed generally lower mean levels when using SPE compared to the combined method (Fig. 7). In the case of the sink, the combined method indicated that the FOG levels varied between 237 $\pm$ 18 to 2023 $\pm$ 441 mg/L compared to 268 $\pm$ 57 and 1640 $\pm$ 1242 mg/L for the SPE method. Although the combined method detected more mean FOG in four of the six samples, variability between triplicates was high, with most of the standard deviation associated with the free oil fraction which varied between 83 $\pm$ 23 mg/L (sample A) and 1569 $\pm$ 462 mg/L (sample D). A marked difference in performance was observed in samples from the dishwasher, from which the combined method extracted significantly more FOG than SPE (between 50 and 300%) from all eight samples, with recorded values ranging between 38 $\pm$ 4 and 477 $\pm$ 64 mg/L; and 24 $\pm$ 4
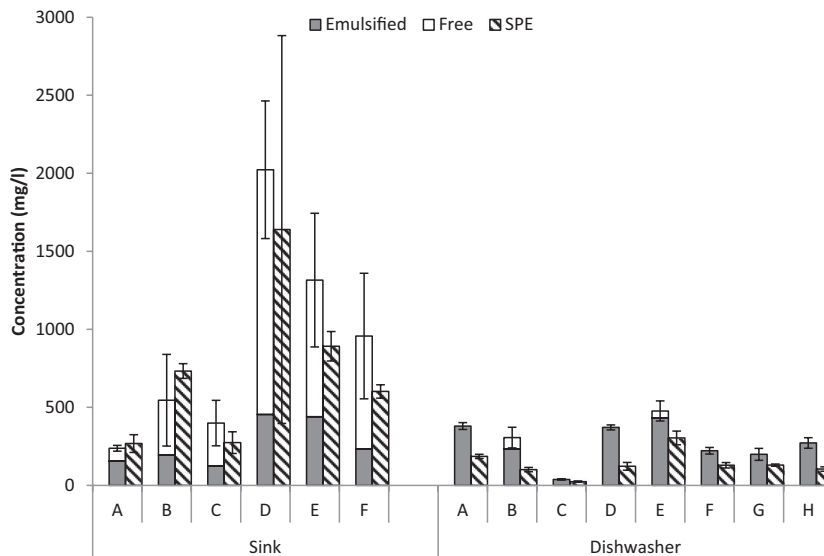

Fig. 7. Comparison of the dual extraction method and SPE for real sink and dishwasher FSE wastewater (*n* = 3) collected over one month. The error is the total emulsified and free oil error for the combined measurement.

to 304 $\pm$ 44 mg/L, respectively. Sample C comprised entirely of emulsified FOG, yet still revealed higher levels than SPE at 38 $\pm$ 4 and 24 $\pm$ 4 mg/L, respectively.

Comparison between the two wastewater sources revealed, that the major difference in the FOG levels were due to differences in the free oil fraction with emulsified levels remaining similar. To illustrate, the emulsified oil concentration in the sink varied between 124 $\pm$ 39 and 454 $\pm$ 79 mg/L compared to between 38 $\pm$ 4 and 432 $\pm$ 36 mg/L for the dishwasher. The current data are consistent with previously reported ranges of between 256 and 1485 mg/L measured across four FSEs (Barton, 2012), 15–256 mg/L for a range of FSEs (Converse *et al.*, 1984), 730–1310 mg/L for four different FSE cuisines (Stoll and Gupta, 1997). However, the current data reveal the level of variation that can occur from sample to sample and due to differences in method. Perhaps more importantly the dual method enables easy separation of free and emulsified oil levels in samples which enables better understanding in relation to treatment in passive gravity separators or with biological additions that can convert the FOG to benign end products.

Conclusions

(1) The development of a modified Gerber method for FOG measurements in FSE wastewater, coupled with a free oil pre-measurement, has been demonstrated to enable more consistent FOG recovery levels than typically observed in the current standard methods.

(2) Furthermore, the addition of a casein precipitation stage has enabled application to non-dairy systems and negated the impact of surfactant on the reliability of FOG measurement in FSE wastewaters experienced in the other methods.

(3) Whilst the LOQ of the new method is higher than standard liquid–liquid extraction techniques it has both excellent recovery and precision down to below the 100 mg/L FOG level making it suitable for discharge monitoring.

(4) The technique is simple, inexpensive and rapid in comparison to standard methods enabling more consistent sampling to be undertaken.

(5) Furthermore, the simple separation of free and emulsified oil contents proposed in this method has the opportunity to greatly enhance insights into management options and support innovation in the sector.

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