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## Sample preparation for thermo-gravimetric determination and thermo-gravimetric characterization of refuse derived fuel



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#### ABSTRACT

Thermo-gravimetric analysis (TGA) is a useful method for characterizing fuels. In the past it has been applied to the study of refuse derived fuel (RDF) and related materials. However, the heterogeneity of RDF makes the preparation of small representative samples very difficult and this difficulty has limited the effectiveness of TGA for characterization of RDF. A TGA method was applied to a variety of materials prepared from a commercially available RDF using a variety of procedures. Applicability of TGA method to the determination of the renewable content of RDF was considered. Cryogenic ball milling was found to be an effective means of preparing RDF samples for TGA. When combined with an effective sample preparation, TGA could be used as an alternative method for assessing the renewable content of RDF.

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#### 1. Introduction

Refuse derived fuel (RDF) is solid fuel manufactured from mixed waste streams. The purpose of RDF is to divert material from land-fills. Municipal solid waste (MSW) and solid recovered fuel (SRF) are related terms. MSW refers to solid waste materials collected by municipal waste management services, which are more or less mixed upon collection. RDF is manufactured from MSW by application of a combination of processes (DEFRA, 2014) which may include: sorting, shredding, hygienization, drying and densification. SRF refers to fuel produced from non-hazardous waste which meets a given set of fuel quality standards like CEN/TS 15357 (DEFRA, 2014; European Commission, 2011).

Energy may be recovered from MSW by directly combusting the material using established technologies such as a grate furnace. This practice is problematic because valuable materials escape recovery and because MSW may contain high concentrations of materials which foul and corrode process equipment (Reichelt et al., 2013; Bankiewicz et al., 2013), or materials which cause pollution control problems. Processing MSW into RDF allows recovery of materials like glass and metals which may be recycled. It may also reduce the concentration of materials which cause maintenance or pollution issues (Chang et al., 1998). Processing may also change the legal designation of the material. In some jurisdictions

processing MSW into RDF allows the material to be shipped to approved facilities outside of the jurisdiction (DEFRA, 2014). Processing also improves the consistency of RDF allowing it to be marketed to consumers of solid fuel as a commodity. However, despite processing efforts RDF is typically quite heterogeneous.

In addition to reducing the amount of material entering landfills, the production of RDF may reduce the overall production of greenhouse gases by displacing conventional fossil fuels. The extent to which use of RDF reduces overall emissions of greenhouse gases depends heavily on what fraction of the fuel is composed of petroleum derived materials (Larsen and Astrup, 2011). Argawal (1988a, 1988b) presented a TGA based proximate analysis technique for RDF and MSW. The technique was informed by the work of Elder (1983) who related experience gained while using a TGA based proximate analysis technique for coal. The advantages of the TGA technique over traditional proximate analysis were the automation provided by TGA and the small sample sizes (Elder, 1983). Argawal noted, that for RDF and MSW, an advantage of the TGA technique was the ability to distinguish between cellulosic materials and petroleum derived materials found in RDF and MSW (Argawal, 1988a, 1988b).

When a dry RDF sample is subjected to a temperature ramp under a flow of inert gas two major mass loss steps are evident. The first of these steps is attributed to cellulosic materials and generally finishes at a temperature lower than 400 °C, and second of these steps is attributed to petroleum derived polymers and it usually begins at temperatures higher than 400 °C and ends around

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500 °C (Argawal,1988a, 1988b; Cozzani et al., 1995; Skodras et al., 2009). After the volatile material is driven out of the sample and only char and ash remain, char may then be determined by switching the gas flow to air. Since, except for polyvinyl chloride, petroleum derived polymers, yield little or no char (Wu et al., 1993; Sorum et al., 2001), the cellulosic fraction may be estimated as the sum of the first, lower temperature mass loss step, and the char yield. The plastic content may be estimated as the mass loss during the second, higher temperature, mass loss step.

Argawal (1988a) considered the ability of TGA to distinguish between cellulosic and petroleum derived materials in RDF and MSW as a major advantage of the technique, because this information could be used to estimate the heating value of RDF and MSW. Concern over global warming has caused governments to employ cap and trade systems to discourage the emission of greenhouse gases. For instance the EU has a cap-and-trade system called the EU Emissions Trading Scheme and, in North America, California has implemented a cap-and-trade system to meet the obligations of the Global Warming Solutions Act of 2006. Such regulations make it important to distinguish between the renewable and non-renewable content of RDF since the renewable portion may be eligible for credit designed to incentivise the use of renewable energy (Muir et al., 2015). Using the information from a TGA proximate analysis of RDF to estimate the renewable fraction of the fuel might be more useful than an estimate of heating value. 14C analysis and selective dissolution are currently used for determining the renewable content of RDF.

<sup>14</sup>C analysis uses the same principal as radio-carbon dating. Cosmic radiation causes the production of <sup>14</sup>C in the atmosphere. From the atmosphere it is absorbed by plants and then animals. As a result of this process biogenic materials contain roughly the same proportion of <sup>14</sup>C as the atmosphere. The uptake of <sup>14</sup>C ceases when life ends and the <sup>14</sup>C in the material decays with a half-life of 5730 years. After about 100,000 years the concentration of <sup>14</sup>C is undetectable. Since fossil materials like coal and oil are much older than 100,000 years they contain practically no <sup>14</sup>C and it is possible to distinguish between modern and fossil carbon by assuming a particular proportion of <sup>14</sup>C in modern biogenic materials (Ariyaratne et al., 2014).

Jones et al. (2013) examined the fossil content of MSW derived solid fuel samples by accelerator mass spectrometric (AMS) detection of <sup>14</sup>C. 30 g samples were combusted in a bomb calorimeter. Carbon dioxide produced by combustion of the samples was collected in sodium hydroxide and then converted to graphite for AMS analysis. <sup>14</sup>C analysis requires assumptions about the amount of <sup>14</sup>C in the biogenic materials found in RDF and MSW. The <sup>14</sup>C concentration in the modern atmosphere has varied significantly over the last six decades. A significant rise in the atmospheric concentration of <sup>14</sup>C occurred during the 1950s and 1960s due to above ground nuclear weapons testing. This rise was followed by a rapid decline caused by international restrictions on nuclear weapons testing (Hua, 2009). The large variation in the atmospheric <sup>14</sup>C over the last six decades means that the proportion of <sup>14</sup>C in the biogenic fraction of RDF and MSW varies significantly.

Fellner and Rechberger (2009) studied the <sup>14</sup>C content in the biogenic fraction of European waste. They found that the average <sup>14</sup>C content of sorted waste was 117.3 percent modern carbon (pMC). The average <sup>14</sup>C content of unsorted waste was found to be 115.2 pMC indicating that sorting tends to select for older materials. The uncertainty, for a 95% confidence interval, was ±3.9 pMC for sorted waste and ±3.5 pMC for unsorted waste and the margin of error for the calculation of biogenic content was at least 6%.

Selective dissolution works by decomposing the biogenic fraction of RDF in a solution of sulfuric acid and hydrogen peroxide then filtering the solution to obtain the fossil fraction (Ariyaratne et al., 2012). Dissolution requires 16 h or >18 h (Schnoller et al.,

2014a). Gram scale samples are used for selective dissolution (Ariyaratne et al., 2012; Severin et al., 2010). The method is complicated by the fact that a portion of the ash forming components in the sample also dissolve in the solution. A separate sample must be used to determine the total ash content, and the ash content of the un-decomposed material, filtered from the solution must also be determined (Ariyaratne et al., 2012). The results of selective dissolution are combined with calorimetry or elemental analysis and results are reported on the basis of heating value (Ariyaratne et al., 2012) or on the basis of carbon content (Ariyaratne et al., 2014).

Small, milligram scale, samples are employed for TGA. Elder considered small sample sizes to be an advantage when the sample availability is limited (Elder, 1983). RDF and MSW sample material is readily available and sample availability should never be a concern. However, the heterogeneity of RDF is a major concern. RDF and MSW are very heterogeneous materials. Samples must be ground to very fine powders to obtain results with reasonably small variances (Schnoller et al., 2014a,b). Producing fine powders from these materials is difficult due to the presence of a variety of materials with different mechanical properties. Even if repeatability is achieved, results may be subject to significant sample preparation error, because sample preparation may alter the composition of the material being analyzed.

TGA has been widely employed for the analysis of RDF and MSW since the proximate analysis technique of Argawal was presented. When TGA is performed on RDF or MSW the focus is usually the determination of kinetic parameters. In some works, samples were ground to 100 mesh and mixed overnight on a rotating riffler (Argawal, 1988a). The same author proposed a method where samples must be ground below 50 μm. Since then samples have been: cooled with liquid nitrogen and milled to fine powders (Cozzani et al., 1995), ground to 40–60 mesh (Lin et al., 1999), milled and sieved to between 150 and 250 μm (Skodras et al., 2009), milled to particle sizes of under 250 μm (Seo et al., 2010), and milled to 150–250 μm (Bosmans et al., 2014). Conspicuously absent from most accounts of laboratory particle size reduction procedures are the amount of material milled, the yield from the milling process, and the equipment used in the milling process.

Despite acknowledged problems with the heterogeneity of RDF and MSW, repeatability is not usually addressed and most procedures seem to be substantially un-validated. When repeatability is addressed, it is usually addressed without reference to variance or standard deviation. Average results from three (Seo et al., 2010) or five (Skodras et al., 2009) replicates have been employed and in some cases the results of duplicate analysis are displayed to show that the variations between results are small (Bosmans et al., 2014)

Any application of TGA to the analysis of RDF or MSW requires the production of fine representative samples. To better understand the effect of sample preparation on TGA of RDF and MSW, in this work a method adapted from Argawal (1988a) was used to analyze materials prepared from commercially available RDF pellets. The materials were prepared using a variety of equipment and procedures. All analyses were performed 15 times to obtain robust estimates of standard deviation. It is anticipated that this information will support the useful application of TGA to the analysis of RDF.

#### 2. Materials and methods

#### 2.1. Refuse derived fuel

Refuse derived fuel pellets were obtained from WastAway<sup>®</sup>. The fuel is produced from MSW by a series of shredding and sorting

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