

TAR FROM BIOMASS GASIFICATION: A SURVEY ANALYSIS METHODS

Marlus Chaves Quintas, marluscq@yahoo.com.br

Department of Mechanical Engineering, Federal University of Pará, Belém – Pará, Brasil, CEP 66075-110

Danielle R. S. Guerra, daguerra@ufpa.br

Department of Mechanical Engineering, Federal University of Pará, Belém – Pará, Brasil, CEP 66075-110

Manoel F. M. Nogueira, mfmn@ufpa.br

Department of Mechanical Engineering, Federal University of Pará, Belém – Pará, Brasil, CEP 66075-110

***Abstract.** Gasification is a technology where biomass can be converted into a combustible producer gas. It is essential that the gas has low concentrations of harmful contaminants for its applicability. Generally, tar and particles into the gas product from different gasifiers as downdraft, updraft, fluidized bed or two-stages, are analyzed by liquid chromatography, gas chromatography and thermogravimetric analysis. In this work, investigations of different analysis methods of particulates and organic contaminants from biomass gasification as well as the measurement data are presented. This survey of analysis methods based in researches founded in the literature showed that the results of gas composition varies with the biomass, sampling collect, and gasifier type. It was observed that the diversity of methods makes any comparison of the tar and particulates data difficult to be analyzed in function of the gasifier operation, this fact showed the necessity of a default analysis method to provide trustful information about the operational quality of the gasifier.*

***Keywords:** sampling, analysis, tar, particles measurement, biomass.*

1. INTRODUCTION

Gasification is an old technology in which there is the production of gaseous by thermal decomposition. Generally the gasification process is used to convert carbonaceous materials in energy or as a basic chemical block for a large number of uses in the petrochemical and refining industries. The materials carbonaceous can be coal, petroleum coke or biomass. The resulting gas from the gasification processes is called syngas that is the product gas mixture which contains amounts of carbon monoxide, carbon dioxide, hydrogen, methane, nitrogen and water. The initial syngas contains particulates and other contaminants such as organic aerosols and condensable hydrocarbons that are the major problems in the producer gas for the application of gasification systems.

The use of the syngas as a source of energy for an internal combustion or other engine requires the gas cleaning, this fact is of great importance. In particular, if particulates and condensable hydrocarbons are not removed they will cause serious problems of deterioration and bad functioning of the engine. The problem of quantity and behavior of tars, condensable hydrocarbons, is very difficult because the gas varies much from one process to another and depends strongly of fuel properties, gasification conditions and reactor type.

The biomass gasification can be processed in different types of technologies and equipments according to the design of fuel bed. The reactors can be classified as fixed-bed, fluidized bed, and entrained flow. The fixed-bed gasifiers are divided in Updraft and Downdraft. In the first one the biomass is introduced at the top of the reactor, the fuel flows down against of the supplied air; the air supplied inlet is at the bottom, the gas product flows in the same direction of the air and leaves at the top of the reactor. In the case that the main use of the gas product is for power production, the major disadvantage is the high quantity of tar in the end of the process.

The second gasifier type, downdraft reactor, the biomass can be introduced from the sides or at the top, it moves in the same direction of the air. The air supply inlet can be through the sides, the gas product flows in the same direction of the biomass and leaves at the bottom of the reactor. Against the updraft gasifiers, the major advantage of the downdraft one is that the gas product has a low tar quantity, which makes this reactor a better solution for engine applications. A schematic view of updraft and downdraft reactors is shown in Fig. 1.

The fluidized bed gasifiers are characterizing by a processes in which the solid behave as a fluid through contact with a gas, in this unit of operation the particles are in suspension. These gasifiers can be divided in bubbling, circulating and atmospheric versus pressurized fluidized bed. When compared with fixed-bed reactors, fluidized bed are compact construction, has complex operation, presents high tar and dust amount into de gas product (Abatzoglou, 2000).

About entrained flow gasifiers, these types generally are used for coal due finer particles size and higher operating temperatures that can be achieved for complete conversion. The fuel particles are pneumatically introduced at the top of the reactor. A schematic view of Fluidized Bed and Entrained Flow reactors are shown in Fig. 2.

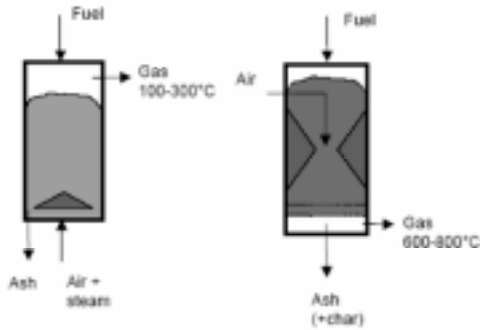


Figure 1. Schematic view of updraft and downdraft gasifiers.

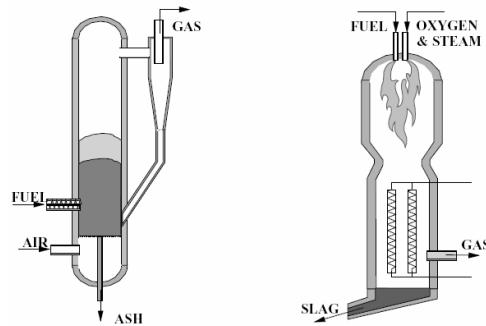


Figure 2. Schematic view of fluidized bed and entrained flow gasifiers.

As mentioned above, the tar associated with the gas product presents a serious problem with regards to its utilization for internal combustion engines. The tar content quantification in the produced gases allows an optimization on the gasifier performance and a better operation condition for the engine.

The aim of this work is to report the different methods and techniques for tar sampling and analysis that have been used to determine the level of particulates resulting of biomass gasification processes. Some results that have been reported in the literature from small scale gasification plants are presented and compared as well as how this problem was discussed by European community and USA.

2. METHODS IN SAMPLING AND ANALYSIS OF TAR

In sampling methods, an important issue is how the sample is extracted from the gasifier. Generally the extraction is done through a heated probe in the isokinetic condition. The heated system is to avoid the condensation of tar compounds. The sampling by isokinetic condition is recommended since tar compounds can form droplets and can then behave like particulates, in this condition the velocity of the sample in the probe is the same as that in the gas stream being sampled.

Methods for integral tar sampling are commonly based on cold trapping using water condensers and cooling traps, occasionally combined with solvent absorption and filters. These methods are based on the EPA-5 method from the early 80's. The adsorption methods were developed in 1996-1998.

The total particulate matter from combustion process can often divided into the soluble organic fraction which is a matter that is extractable using an organic solvent, and the insoluble fraction which remains after extraction (Hindsgaul, 2000). For trapping the tar the most common solvents have been dichloromethane, cyclohexane and acetone. The separation of the soluble organic fraction and the insoluble fraction depends on the solvent used. However, dichloromethane is in very widespread use as a solvent for total particulate matter because it is known to dissolve all tar compounds well.

The methods founded in the literature for tar and particle collection is based on condenser and distillers equipments. The methods are impinger bottles or Petersen column. The first one consists of a series of impinger bottles in which water and tar are condensed from the process gas by absorption in a solvent. The heat released by gas cooling and condensation is removed either in an external water bath or by an additional heat exchanger before the condenser. The condenser can be a standard impinger bottle or optionally be equipped with an internal liquid quench system which is especially suitable for producer gases containing higher tar levels. Figure 3 shows a set-up example of the impinger bottles.

The other known method, Petersen column, was developed as alternative equipment for the impinger bottles. The equipment consists of two washing stages filled with a solvent, the first is a washing stage with an impinger, and the

bottom of second stage consists of a G3 glass frit. The two washing stages are filled with the washing solvent. The equipment is jacket cooling. The column must be constructed thus the replace of the glass frit is easy if it is polluted by particles. The Petersen column is shown in Fig. 4.

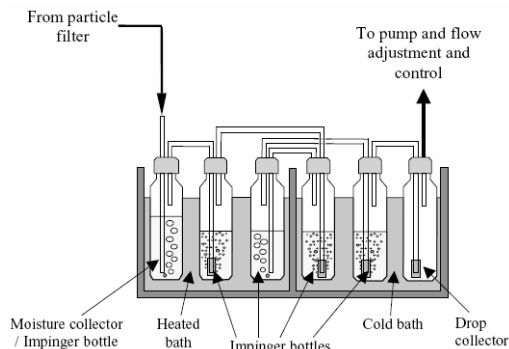


Figure 3. Example of the impinger bottles set-up.

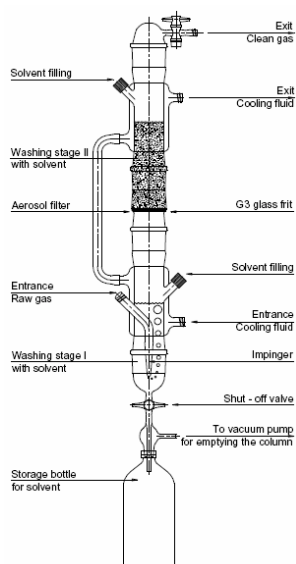


Figure 4. The Petersen column.

The amount of condensate deposited into the impinger bottles will unavoidably be a function of the cooling conditions as temperature and gas flow rate used. The condensate may be treated and analyzed for its composition determination by appropriate techniques as thermogravimetric, scanning electron microscopy, gas chromatography and liquid chromatography. The method of analysis to be applied is dependent on the way of formation and the content of the tar substance to be determined. A briefly description of some usual methods is presented in the following paragraphs.

Thermogravimetric analysis (TGA), is an analytical technique used to determine the thermal stability of a material and its fraction volatile components by monitoring the weight change that occurs as a specimen is heated. The measurement is normally carried out in air or in an inert atmosphere, such as Helium or Argon. In this technique the weight is recorded as a function of increasing temperature. In addition to weight changes, some instruments also record the temperature difference between the specimen and one or more references (differential thermal analysis - DTA), or the heat flow into the specimen compared to that of reference (differential scanning calorimetry - DSC). The method does not provide information about chemical composition. Usually in TGA, the loss of mass during combustion is interpreted as the content of pure carbon structures and the residual mass is interpreted as the ash content.

To examine geometries and size of particles, the scanning electron microscopy (SEM) can be used. SEM permits the observation and characterization of heterogeneous organic and inorganic material on nanometer to micrometer scale. In the instrument, the area to be examined or the micro volume to be analyzed is irradiated with a finely focused electron beam. The signals are obtained from specific emission volumes within the sample and can be examine many

characteristics of the sample as surface topography, crystallography and composition (Goldstein, 1981). The signal of great interest is the secondary and backscatter electrons.

To obtain a rough overview of which chemical groups could be present in the samples, the analytical method is chromatography that is applied to separate the compounds into fractions based on their polarity. Chromatography process can be defined as separation techniques involving mass-transfer between stationary and mobile phases.

Gas Chromatography is a type of chromatography in which the mobile phase is a carrier gas, usually an inert gas such as helium, nitrogen or hydrogen and the stationary phase is a microscopic layer of liquid or polymer on an inert solid support, inside glass or metal tubing, called a column. An example of this system is shown in Fig. 5.

Gas chromatography coupled with mass spectrometry detection (GC/MS) is a well established analytical method for identification and quantification of single compounds. With regard to the application for tar compounds, GC/MS is mostly used for the analysis of benzene aromatic compounds. This may be the result of working definitions for tar that emphasize the importance of the share of aromatics in tar samples from gasifier systems, such as in the definition given in Milne et al. (1998). However, primary tar mainly contains oxygenated compounds with various structures, both aromatic and aliphatic, such as the classes furans, acetones, aldehydes etc. The quantitative analysis of these oxygenated primary tar compounds by means of GC/MS is rarely reported in the literature. Some studies on the characterization of fast pyrolysis oils contain corresponding analysis. Commercial GC can be finding in two detectors versions, thermal conductivity detector (TCD) and flame ionization detector (FID).

The liquid chromatography (LC) utilizes a liquid mobile phase to separate the components of a mixture. These components (or analytes) are first dissolved in a solvent, and then forced to flow through a chromatography column under a high pressure. In the column, the mixture is resolved into its components. The amount of resolution is important, and is dependent upon the extent of interaction between the solute components and the stationary phase. The stationary phase is defined as the immobile packing material in the column. The interaction of the solute with mobile and stationary phases can be manipulated through different choices of both solvents and stationary phases. As a result, LC acquires a high degree of versatility not found in other chromatographic systems and it has the ability to easily separate a wide variety of chemical mixtures.

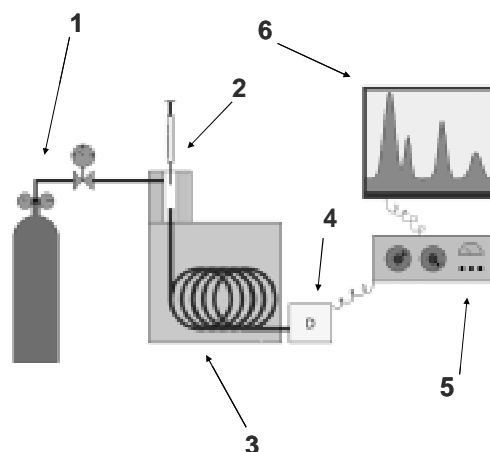


Figure 5. Gas chromatography system: 1) gas reservoir, 2) sample injector, 3) Column, 4) detector, 5) amplifier signal, 6) data register.

A wide number of tar sampling and analysis methods has been reported in the literature. In this work only a short overview of principal methods is shown.

3. RESULTS

The biomass gasification provides tars with complex heterogeneous mixture of organic molecules as aromatics, phenols, bases, asphaltenes, preasphaltenes and particulates matter, which their concentration is related to the formation conditions as temperature, pressure and reactor design.

A characterization of wood-derived tars was presented by Beall and Duncan (1980). In this investigation various solvent systems for tars from four wood gasifiers in Canada were used. The results have shown that the best mixture was ether, tetrahydrofuran and ethanol. The tars were fractionated by column chromatography into hydrocarbons, aromatic hydrocarbons and benzofurans, ethers, nitrogen compounds and hydroxyl compounds.

Aiken et al. (1983) founded that acetone was the best solvent for sample recovery and cleanup of sampler components. In this analysis, the acetone is evaporated at 30°C to weight tar residue. The research results gave tar at 6.1wt% of feed, particulates at 0.4, and condensate at 0.3.

Leppälahti and Kurkela (1991) have measured tar and N-compound formation from peat in laboratory scale, atmospheric pressure fluidized bed gasifier. Effects of secondary air in the freeboard and dolomite in the bed were measured. The gas was extracted by a stainless-steel sample probe maintained at 250°C to prevent tar condensation.

After the extraction, tars were condensed in wash bottles at +5°C and -70°C with dichloromethane. The gas was analyzed through chromatography. The results shown that benzene, toluene and naphthalene were major tars under most conditions.

Bui et al. (1994) have been reported the average tar content in the gas product from wood gasification in range from 2g/Nm³ for conventional downdraft gasifiers to 58g/Nm³ for conventional updraft gasifiers. Brown et al. (1986) and Bridgwater (1995) have suggested different desirable levels of tar quantity in the range of 10 to 50mg/Nm³. For engines, tar and dust loads must be lower than 10mg/Nm³. In this investigation a throatless two-gasifier was used. The tar was collected in ice water. As result the gas product presented a tar reduction from 3600mg/Nm³ to 92mg/Nm³.

Characteristics of tar from wood pyrolysis in a fixed-bed reactor, was investigated by Brage et al. (1996). In the experiment tar was collected in a series of traps including ice-acetone and dry-ice acetone. Total tar was determined gravimetrically from the crude tar extract by rotary evaporation of the solvent to constant weight at 50°C and 2kPa. As result, tables of major condensable products are given.

Breage et al. (1997) proposed a method to reduce sampling and sample separation time based on solid-phase adsorption on amino phase. The method is suitable for intermittent trapping of tar compounds ranging from benzene to coronene present in product gases from thermal decomposition of biomass at 700 to 1000°C. Experiments were performed in a pressurized free-fall tubular reactor. The sampling was carried out of one to three samples per minute and compared with one or two samples per hour using the conventional cold trapping technique. In this study the gas analysis was determined by gas chromatography with flame-ionization detection. The authors have been shown that the method provided a much faster and accurate alternative to traditional cold trapping methods.

Vural (1997) analyzed the activity of fluidized bed particles towards the volatile matter of bituminous coal by the measurement of tar and CO₂ concentration. Thermogravimetric analysis of the fluidizing particles was performed indicating that calcium-containing particles exhibited the largest weight loss and a mass spectrometer was connected to the thermogravimetric instrument to identify the gases responsible for the weight loss.

Bhattacharya et al. (1999) presented results of an experimental study on two-stage wood gasification to reduce the tar content of the gas. In their study, for a particular primary air flow rate, an increase in the secondary flow of the two-stage gasifier resulted in decrease of the tar content and the CO₂ and H₂ concentrations while that of CO increased. The results shown the tar content was in the range 19 – 34mg/Nm³ for a charcoal gasifier coupled to a two-stage wood gasifier. The study presented the effects of the air flow rate on the gasifier performance, the effects of moisture content and wood species on tar quantity.

Hindsgaul et al. (2000) investigated the physical and chemical characterization of particles in producer gas from wood chips in a two-stage downdraft gasifier. The majority of the total particulate matter mass was identified using scanning electron microscopy. 77% of the total particulate matter was determined by thermogravimetric analysis which results have shown to be carbon structures. The dichloromethane-soluble fraction, corresponding 11% of the total particulate matter, was extracted, separated into fractions of varying polarities using adsorption column chromatography and analyzed using gas chromatography with a flame ionization detector. The particle collection was carried out by a cascade impactor device.

There are two problems in the investigation of tar, one is that there still no consensus among researchers in what compounds found in the product gases of a gasifier make up tars or not. The term tar is generically used to designate residues produced in the thermochemical conversion of organic materials. In the context of biomass gasification, tars have often been operationally defined as the condensates deposited in equipment downstream of the gasifier. Tars could thus be identified as those compounds that make difficult the usage and handling of the producer or synthesis gas.

Another problem encountered, is the absence of standards regarding the methods for sampling and analysis. Not only have a large number of different tar sampling methods been developed, also different quantification methods have been used. Since 1998 the members of the IEA Bioenergy Gasification Task have tried to bring some standardisation in this field and some researchers have been published in this subject (Abatzoglou et al., 2000), (Moersch et al., 2000), (Knoef, 2000).

The VTT Energy, Technical Research Centre of Finland, in 1998 has been presented their standard methods for sampling and measurement contaminants from gasification process. The technical report was a contribution to the Thermal Gasification Biomass Activity of the IEA Bioenergy Agreement.

In the beginning of the year 2000 the IEA Bioenergy Gasification Task, the UsDoE and DGXVII of the European Commission published their definition of relevant tars in gasification and sampling and analysis methods (Simell, 2000) and (Technical Report, 2005). These might in the future attain the status of internationally accepted standard methods.

Full quantification of tar components is a difficult, expensive and time consuming task, these facts together with the wide number of sampling and analysis methods make any comparative study of tar nature to be difficult. Beside these, many experiments were run using a large variety of reactors type functioning in different conditions and with different biomass as fuel. As results of this review work, some data provided in the literature are shown in Table 1. The data had been organized in accordance with the objective of the present study to shown the variety of gasifiers, the used fuel, the sampling methods, the analysis methods and the result obtained.

Table 1. Data results provided in the literature.

Reference	Gasifier type	Biomass	Sampling method	Analysis method	Product detected
Oesch et al. (1996)	Pressurized fluidized bed	Pine sawdust, pine bark, forest residues, wheat straw, preat, polish coal	Filter, cooled tube, condenser and impinger bottles	Gravimetric, gas chromatography, gel permeation chromatography (GPC), analytical pyrolysis combined with elemental detection	Molecular weight of organic compounds in GPC
Narváez et al. (1996)	Atmospheric bubbling fluidized bed	Pinus pinaster sawdust	System similar to the one used in VTT; five impinger flasks or traps of 200cm ³ each	Total Organic Carbon Dohrmann Analyzer DC90, GC-TCD and GC-FID	With an equivalence ratio of 0.30 the tar content was between 4 and 18g/Nm ³
Gil et al. (1997)	Fluidized bed	Pine wood chips	System similar to the one used in VTT at Espoo (Finland)	Dohrmann Analyzer DC90, GC	Gas composition and tar content of 5g/Nm ³ ; its main components are phenol, cresol, naphthalene, indene and toluene
Brage et al.(1997)	Pressurized fluidized bed	Birchwood and birchwood char	Cold trapping, solid-phase adsorption, solid-phase micro extraction.	GC-FID	Phenol, o-Cresol, m-Cresol, Benzene, Toluene, p-Xylene, o-Xylene, indene, naphthalene, 2-methylnaphthalene, 1-methylnaphthalene, acenaphthylene, Fluorene, Phenanthrene, Anthracene, Pyrene. The quantity of these compounds was different for the three sampling techniques.
Gómez et al.(1999)	Fluidized bed	Bagasse pellets from sugar cane	Sample conditioning using on-line filters	GC	Gas composition and ash
Bhattacharya et al. (1999)	Two-stage	Wood-chip	Electronic heater, ceramic wool filter, counter-flow glass condenser	Two on-line gas analyzers	Tar content was 19 – 34 mg/Nm ³ (without a floating-drum gas-storage system).
Hindsgaul et al.(2000)	Two-stage downdraft	Wood chips	Tubular fiber filters, membrane filters and cascade impactor	Scanning electron microscopy (SEM), TGA, GC-FID, Extractions	Total particle concentration was 270mg/Nm ³ .
Moersch et al.(2000)	Fluidized bed and fixed bed	Wood	Condensation on a filter	New analyzer prototype with FID detector	Tars content between 200 and 20,000mg/Nm ³ . Benzene, toluene, Other BTX, phenols, PAH, Heavy tars
Brage et al. (2000)	Pressurized fluidized bed	Birch and Daw mill coal	Solid phase adsorption	Liquid chromatography, GC-FID, GC-TCD	Major tar compounds was benzene, toluene, indene, naphthalene, phenol
Padban et al. (2000)	Pressurized bubbling fluidized bed	Bark and sawdust from forestry residues	LU Tar Sample, cold trapping; combined LU-cold trapping; solid phase absorption (SPA)	Gravimetric measurements and GC-MS	Light PAHs

Bhattacharya et al. (2001)	Multi-stage	Coconut shell	Electronic heater, ceramic wool filter, counter-flow glass condenser.	GC-online, infra-red gas analyzer and a thermal conductivity gas analyzer	The lowest tar content found was about 28mg/Nm ³ .
Adegoroye et al.(2004)	Spouted bed	Sewage sludge	Cooled filter	GC-FID, GC-MS, TGA, Ultra violet fluorescence spectroscopy (UV-F) and Size Exclusion Chromatography (SEC)	Aromatics and polycyclic compounds with heteroatom substitution such as phenanthrene, fluorine, chrysene, phenol, indole, quinoline, Pyrene and naphthalene (by GC/MS). Aromatics with molecular weights up to 300 and aliphatics up to 500 (by GC/FID). Presence of higher concentrations of larger polynuclear aromatic ring systems, heterocyclic structures and alkyl or heteroatom substituents as the temperature of tar formation increased (by UV-F).
Li et al.(2004)	Circulating fluidized bed	Cedar, cypress, hemlock, SPF and PS	Tar Protocol	GC-TCD	Tars contain about 78% Carbon, 6% Hydrogen, 0.7% Nitrogen, 12% Oxygen, less than 0.5% Sulphur.
Paasen and Kiel (2004)	Bubbling fluidized bed	Woody	The Guideline method	GC, TGA and HPLC	Alkyl-substituted tar compounds, heterocyclic tar compounds and PHA (primary, secondary and tertiary tars)
Cao et al. (2006)	Fluidized bed	Mixed wood sawdust	A water cooler and an ice trap in series.	GC- TCD and FID	The tar content varies in function of the temperature. The concentrations of light hydrocarbons in fuel were presented.
Qin et al.(2007)	Pressurized fluidized bed	Heartwood and sapwood	Cooled tube and small Soxhlet	Gel permeation chromatography, photodiode array detector and Ultraviolet	Molecular weight distribution, gas concentration.

3. CONCLUSION

Different sampling and analysis methods have been used by manufactures, researchers and others workers to determine the content of particulates and organic contaminants in the gas product from the biomass gasification process. The diversity of these methods represents a barrier to the further development and commercialization of the gasification technology, just because the efficiency of the gas is fundamental to the successful operation of power generation based on the gasification.

This preliminary work provides an overview on how much the researchers are placing its efforts to develop a standard procedure to guarantee that the gasification reactor will be efficient. It was noted that independently of the reactor type, of the biomass as fuel and the conditions of work, the quantity of particulate and tar produced in the process seemed to be a better parameter to characterize the reactor efficiency.

Although all efforts, the protocol proposed by the members of the Gasification Task of the IEA Bioenergy Agreement, has some limitations and requires some adjustments. In Brazil there are two research institutions that have a system similar to that proposed by IEA, but no efforts is seemed to promote or investigate a sampling method.

5. ACKNOWLEDGEMENTS

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