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Research article

Microwave plasma application in decomposition and steam reforming of model tar compounds

P. Jamróz[a], W. Kordylewski[b], M. Wnukowski[b,*]

[a] Wroclaw University of Science and Technology, Faculty of Chemistry, Poland
[b] Wroclaw University of Science and Technology, Faculty of Mechanical and Power Engineering, Poland

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ABSTRACT

Plasma methods can be considered as a novel and efficient way of tar disposal from process gases. In this lab-scale study, a microwave plasma reactor has been developed and tested for the conversion of tar surrogates, i.e. benzene, toluene, and 1-methylnaphthalene, in a nitrogen stream. The effect of tar concentration, gas flow rate and steam addition on conversion effectiveness has been analyzed. It was demonstrated that the process efficiency can be as high as c.a. 98% with the initial tar concentration of 10 g/Nm^3, the nitrogen gas flow rate being 30 L/min and the steam-to-carbon ratio equal to 3. The conversion efficiency decreased with increasing tar concentration and gas flow rate. At the same time, the increase in the steam addition significantly enhanced the conversion rate. It has been revealed that the main products of the tar model compounds plasma conversion were: acetylene, soot, benzene derivatives (e.g. benzonitrile, phenylethyne, naphthalene and others) and cyanides. Their amounts were significantly decreased in favor of CO, CO_2, and H_2 by steam addition. Additionally, optical emission spectroscopy has been applied for the purpose of microwave plasma diagnostic and identification of the reactive species in the plasma zone. Moreover, a quantitative analysis of the main gaseous and aromatic byproducts has been conducted by means of gas chromatography.

1. Introduction

Gasification can be considered as one of the most flexible fuel conversion processes. The process gas can be utilized in boilers, engines, turbines or even fuel cells to produce energy. Moreover, it can be converted into syngas, which might be applied in chemical syntheses of many products, including components of liquid fuels like alcohols or hydrocarbons. Another advantage of gasification is that almost any carbonaceous material can undergo this process i.e. biomass or wastes. This is essentially important in terms of environmental policy and renewable energy sources. These combined advantages of the gasification process explain the attention it has attracted in recent years. In practice, however, the utility of the process gas can be strongly limited by the gas quality, because many impurities, such as ash, volatile alkali metals, acid gases, and tars are generated during gasification.

While there are a few definitions and categories of tar [1], it can be described as a mixture of heavy organic compounds – mainly of aromatic nature. These compounds tend to condensate in low temperatures and/or elevated pressure. As a consequence, they may cause malfunctions of turbines, engines and fuel cells as well as fouling and blocking of pipelines and filters [1]. Therefore, a firm, proven and effective technique of the tar content reduction in the process gas is essential for the wide commercialization of biomass gasification.

Many methods have been developed to reduce the amount of tar in the raw syngas. Primary methods focus on limiting tar concentration using the gasification process optimization i.e. process temperature, gasifier design, gasifying medium etc. [2]. While these procedures are essential and should be first to consider and obligatory in terms of tar control, they are usually insufficient to reduce the tar concentration to the desired level. It is stated that the permissible tar concentration is about 100 mg/Nm^3 for reciprocating internal combustion engines and even less for gas turbines or synthesis purpose [3,4]. To achieve these numbers secondary methods must be introduced. Among them, four groups can be derived: mechanical, thermal, catalytic and plasma methods [1,5].

Mechanical methods dominate in pilot and commercial scale installations [6]. However, they often show low efficiency in tar removal [7]. Although progress has been made, resulting in a high-efficiency cleaning technology like OLGA system [1,8], the mechanical methods still have several major drawbacks. Firstly, application of these methods results only in the removal of tars, not their conversion. This means that the tar problem is only moved away (as the tars are absorbed or

* Corresponding author.
E-mail address: mateusz.wnukowski@pwr.edu.pl (M. Wnukowski).

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adsorbed), rather than being completely solved [3]. Secondly, mechanical methods remove mainly heavy compounds. Volatile components (e.g. benzene or lighter compounds) can relatively easily pass the cleaning device. While this phenomenon can be accepted in case of syngas application in IC engine, it can remarkably reduce syngas potential in Fischer-Tropsch or methanol syntheses [9]. Thermal methods lack these disadvantages but are highly inefficient. It has been proven that decomposition of an aromatic compound, like benzene or naphthalene, requires a temperature above 1100 °C and a residence time from 0.26 to 10 s (depending on the applied temperature) to achieve high efficiency [10–12]. Obtaining such high temperature is problematic considering energy efficiency and used materials. Significantly lower temperatures are required for catalytic conversion methods [1]. Additionally, due to reforming of all organic compounds, these methods allow achieving higher yields of valuable hydrogen and carbon monoxide. This process can be enhanced even further with steam reforming [13,14]. These advantages attracted a great attention to catalytic methods in recent years. Many materials have been tested as possible catalysts, e.g. metals (mainly nickel), biochar, natural minerals and many others. Description of these catalysts and their performance in tar conversion can be found in many detailed review articles [15–17]. However, catalytic methods are not flawless. The most fundamental drawbacks are: the catalyst cost, its lifetime, poisoning due to sulfur compounds and carbon deposit and a resulting efficiency reduction that comes with time [18,19].

Plasma methods, on the other hand, lack these basic drawbacks. Although they seem to be the most costly and technically advanced methods, alike catalytic methods, plasma methods show high conversion rate of hydrocarbons (including tars) and enhanced production of hydrogen and carbon monoxide [1,19–21]. The high efficiency of plasma is a result of its high temperature and presence of reactive species i.e. electrons, ions, radicals etc. that enhance the chemical reactions. Since plasma can be obtained by many means there are several techniques that have been implemented in tar decomposition. Corona discharge plasma [22,23] and gliding arc plasma [24,25] seems to be the first that were thoroughly investigated in terms of tar destruction. With time, the latter one has received greater attention being a subject of many works [19,20,26]. Yet, there are other solutions that might be an interesting and efficient alternative, for ex.: arc plasma [27], dielectric barrier discharge (DBD) [28], and microwave (MW) plasma [29,30].

From all the mentioned plasma technologies, microwave plasma shows some properties that make it particularly interesting and promising in the context of tar conversion. First of all, in contrary to most plasma methods, microwave plasma is an electrodeless one. Electrodes are the factor that strongly limits the application of plasma due to their erosion [31,32]. The erosion can be a particularly problematic issue in the presence of oxidizers (like oxygen or steam) or particles [32] which are present in the raw syngas. Secondly, plasma technologies may require an advanced and complicated power source that is designed and manufactured specifically for the purpose of plasma set-up [22]. On the other hand, magnetrons used in microwave plasma are the same that are used in other industrial applications, e.g. drying or food processing. They are relatively cheap and have a simple and compact construction similar to the domestic MW ovens [33]. Moreover, they are produced by many companies and their power can vary from few to hundreds of kW, giving a potential for scaling up of the technology. Lastly, but maybe most importantly, in microwave plasma, a great amount of energy is distributed into vibrational excitation [33]. This physical phenomenon is essential in the chemistry of compounds like CO, CO_2, H_2, and N_2, all of which can be present is syngas [34]. It has been proven that MW plasma is an effective method of conversion of light organic compounds [35] and carbon dioxide [36], resulting in a high content of the most valuable products: carbon monoxide and hydrogen. Despite these advantages, only a few works have been devoted to investigating this method [29,30]. Although they have presented microwave plasma as a promising method for tar decomposition, there is still a great lack of knowledge about the microwave plasma potential in this matter. Essentially, to our knowledge, there is no work that would investigate typical tar surrogates, e.g. benzene or toluene decomposition in pure nitrogen microwave plasma. These conditions are crucial, due to the fact that nitrogen plasma is much closer to the real producer gas atmosphere rather than air plasma [29] or argon/nitrogen plasma [30]. Introducing the aforementioned aromatics allow conducting reliable and repeatable experiments which can be compared with other research. At the same time, these compounds show a high thermal stability and, in fact, they are the basic components of tar formed during biomass gasification.

The purpose of this work is to expand the knowledge of the microwave plasma potential as a method of syngas conditioning. For this purpose, a series of experiments with the use of nitrogen microwave plasma and basic tar compounds, i.e. benzene, toluene, and methylnaphthalene have been conducted. The paper is focused not only on the decomposition efficiency, depending on tar substitute concentration and gas flow, but great attention is also given to the products obtained due to tar decomposition.

2. Experimental

2.1. Experimental setup

A schematic diagram of the experimental set-up is presented in Fig. 1.

During the extended experiments, two MW plasma reactors were used. The first one, used in research on biomass and plastics pyrolysis and gasification, wasn't specifically designed for tar conversion. Its construction resulted in some technical obstacles, e.g. no possibility of optical emission spectroscopy (OES) analyses, inconvenient products sampling and steam introduction, problems with plasma stability. Thus, it was only used to carry out preliminary research to investigate the method's potential. The analyses of the model tar concentration (benzene or toluene) and the gas flow influence on the decomposition efficiency were carried out with the use of the first reactor. Being proven that the reactor has been able to decompose tar with a high efficiency, the second reactor was designed and manufactured for main experiments. There were two major differences between the reactors. The first one had the microwave power of 1200 W, and the second of 1800 W. The second difference was the reactor's quartz tube dimensions: 85.5 mm height and 21.5 mm inner diameter in the first reactor and 600 mm height and 25 mm inner diameter in the second reactor. Despite these differences, the most important elements of the reactors, and so the principle of their work, remained identical to any other microwave plasma reactor [29,35]. The MW plasma reactors consisted of 2,45 GHz microwave generator (A) connected to power supply, a circulator (B) for preventing the generator's destruction by recurring microwaves (B), a reflectometer (C) for measuring the microwaves power and a waveguide with a movable plunger (E). The MW plasma was ignited in a quartz tube that was fixed in the waveguide.

Nitrogen (technical purity 99.5%) was used as a plasma agent and carrier gas at the same time. In the case of preliminary investigations, focused on the conversion efficiency in correlation to the gas flow rate and benzene/toluene concentration, the tar compound was introduced from a three-neck flask (H) by the carrier gas. In the main research, a continuous injection of the aromatic compounds into a heater (heated up to 350 °C) was provided by a peristaltic pump (Ismantec, Reglo ICC) (F). In the heater, the compounds were vaporized and mixed with the stream of nitrogen and/or steam (when used). A similar procedure was applied in the case of steam introduction. The temperature of the mixture flowing into the reactor's head was c.a. 230 °C allowing the tar compound and water to be kept in a gaseous state.

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