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Assessment of Heavy Metal Content in Plant Waste and Environmental Impact Evaluation through Thermal Analysis

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Abstract

The growing trend of converting plant waste into energy requires a comprehensive analytical investigation into the potential environmental risks of heavy metal pollution. Therefore, this paper presents an in-depth analytical study of the heavy metal content in waste samples of Eucalyptus collected from three locations in Iraq: Al-Khadra District, Al-Taji, and Samarra. The analysis included ultimate and proximate, ICP-MS, Thermal, and SEM-EDX analysis. TGA/DTG analyses were used to characterize the thermal behavior of the samples; the obtained TG and DTG curves were used to evaluate the volatilization of heavy metals during the gasification process. Results indicated that the samples followed a general trend of heavy metal contamination with Pb, followed mainly by Al, Fe, Ni, As, and Cu. Increasing temperature through TGA experiments raised the heavy metal content in char to maximum volatilization at 900°C. This work emphasizes assessing heavy metal content in plant waste, intending to evaluate the environmental impact of such wastes and has shown how gasification is among the promising thermos-conversion technologies for waste-to-energy conversion. The outcome of this work adds to the development of strategies that could be used to mitigate the environmental impact of heavy metal contamination in plant waste while assisting in sustainable energy production.

Keywords: Heavy metal contamination, Plant waste, Environmental assessment, SEM-EDX, TGA/DTG.

تقدير مستوى المعادن الثقيلة في المخلفات النباتية ودراسة التأثيرات البيئية بالتحليل الحراري

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الخلاصة

تتطلب التوجهات المتزايدة نحو تحويل المخلفات النباتية إلى طاقة إجراء تحقيقات تحليلية شاملة حول المخاطر البيئية المحتملة الناتجة عن تلوث المعادن الثقيلة. لذا، تقدم هذه الدراسة تحليلاً معمقاً لمحتوى المعادن الثقيلة في عينات مخلفات الكينيا (الإيكالبتوس) المجمعة من ثلاثة مواقع في العراق: حي الخضراء، والتاجي، وسامراء. شمل التحليل الدراسة النهائية والتحليل التقريبي، وتحليلICP-MS ، والتحليل الحراري، بالإضافة إلى تحليل المجهر الإلكتروني الماسح مع الطيف التبعي للأشعة السينية .(SEM-EDX) استُخدم التحليل الوزني الحراري (TGA) ومشتقاته (DTG) لتوصيف السلوك الحراري للعينات؛ حيث استُخدمت

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منحنيات TG و DTG المستحصلة لتقييم تطاير المعادن الثقيلة خلال عملية التحويل الغازي. أشارت النتائج إلى أن العينات اتبعت نمطاً عاماً للتلوث بالمعادن الثقيلة يتصدره الرصاص(Pb) ، يليه كل من الألمنيوم (Al)، والحديد(Fe) ، والنيكل(Ni) ، والزرنيخ(As) ، والنحاس .(Cu) أدت زيادة درجة الحرارة خلال تجارب TGAإلى رفع محتوى المعادن الثقيلة في المتبقي الكربوني (الفحم) حتى وصل إلى أقصى درجة تطاير عند 900°م. تسلط هذه الدراسة الضوء على أهمية تقييم محتوى المعادن الثقيلة في المخلفات النباتية بهدف تقييم الأثر البيئي لهذه المخلفات، وتوضح كيف يُعد التحويل الغازي من بين تقنيات التحويل الحراري الواعدة لتحويل المخلفات إلى طاقة. تسهم نتائج هذا العمل في وضع استراتيجيات يمكن استخدامها للتخفيف من الأثر البيئي لتلوث المعادن الثقيلة في المخلفات النباتية، مع المساعدة في تحقيق إنتاج الطاقة المستدامة.

1. Introduction

There is growing concern about the anticipated rise in the global population, which is expected to lead to increased waste generation and associated environmental and public health issues [1, 2]. Most importantly, the incineration of waste contributes to exponential worsening of environmental and public health issues [3]. Moreover, the provision of food and energy sources has become a virtually unattainable goal, and, hence, sustainable strategies for producing energy have become indispensable for tackling these issues. Among those material efforts, a part of it such as from waste to energy is likely to raise socio-ecological concerns [4]. However, the current use of biomass for power generation has an environmental impulse from its small-scale environmental footprint as thermal conversion is not increasing CO_2 release in the atmosphere [5-7]. The occurrence of heavy metals in plant-based wastes has recently become an important environmental concern, especially when the waste is thermally converted to energy [8, 9]. Thermal processing may facilitate the release of heavy metals into the environment, posing potential risks to ecological integrity and human health [10, 11]. Previous studies on biomass conversion have been mainly directed towards the efficiency of biomass conversion and energy output; less attention has been given to the fate and behavior of heavy metals in the process [12, 13]. Understanding the transformation and migration of heavy metals during thermal conversion is a key issue in the development of environmentally sound waste-to-energy solutions, particularly in highly industrialized areas or those with high traffic density, where high levels of heavy metals from environmental exposure can potentially accumulate in plants [14, 15]. At the same time, many analytical techniques have been used to investigate biomass characteristics, such as HPLC [16], Nanotechnology [17], mass spectrophotometry analysis (MS) [18], GC/MS [19], FTIR [20], microwave digestion [21] and Thermal analysis [22]. Therefore, comprehensive studies that combine multiple analytical approaches to understand both heavy metal content and thermal behavior of plant waste materials are still needed. Several longitudinal studies have reviewed biomass as an energy source [23, 24]. Other areas of study include environmental impacts [25], metal content and its fate in biomass [26], and characteristics through thermogravimetric kinetic studies [27-29]. In Iraq, research on heavy metal concentrations in plant waste has been limited, as most studies in the region have focused instead on analyzing vitamin content [30] and pharmaceutical compounds [31-33].

Thermal characterization methods such as TGA, DSC are used in this research to associate with plant waste for its thermal stability and compositional behaviour under controlled temperature. These approaches could provide valuable information about the behavior of organic matter, moisture levels, as well as the presence of inorganic by-products influenced by contamination with other compounds. The findings of this study can provide a tool allowing the linkage of thermal degradation stimuli with the pollutants responsible and the effects on the plant's cellular structure sustainability and the potential release of the same during the decay of plant material. Therefore, the aspects outlined should also be included in risk assessments and the remediation measures developed. The use of thermal analysis greatly enhances the capability of the present work to correlate pollution exposure with material changes in bioindicators such as leaves from eucalyptus.

The major goal of this work was to assess the amount of heavy metals in the plant waste, especially Eucalyptus, for ascertaining the biological impact via the thermogravimetric methods of investigation. This research dealt with a subject not previously studied in the literature of Iraq, namely, the relationship between the contamination in plant waste and the parameters of the decomposition reaction. In the present investigation employed composite methods of the investigation of the samples collected from three places in Iraq, the Al-Khadra district, Al-Taji, and Samarra. The analytical methodology included standard methods for determining calorific value, chemical characterization by proximate analysis, and advanced instrumental techniques, such as SEM-EDX for elemental analysis and TGA for thermal characteristics under gasification conditions.

2. Materials and methods

2.1. Materials

All the materials used in this work, including Nitric acid (HNO_3) and hydrogen peroxide (H_2O_2), were of analytical grade and supplied by Sigma-Aldrich.

2.2. Methods

2.2.1. Collection of the Eucalyptus waste plant samples

Samples were obtained from three highway-adjacent sites in Iraq—specifically the Al-Khadra District (Baghdad), Al-Taji, and Samarra, at 30 cm above ground level, precisely at 11:00 a.m. during May 2024. Samples from these sites were taken solely because they were believed to have high contamination due to several pollution contributors: emissions from locally located power generators, highway traffic, and nearby industries.

2.2.2. Preparation of the Eucalyptus samples

Samples underwent a three-step preparation process. First, they were washed with tap water to remove physical contaminants such as dirt and sand. Next, the samples were air-dried and dried at 40 °C ±5 °C for one hour. Finally, the dried Eucalyptus plant material was ground into a fine powder [34].

2.2.3. Extraction and determination of heavy metals

Two separate analytical methods were applied, one focused on the Eucalyptus plant and the other on the char generated from Eucalyptus gasification. For the metal(loid) analysis in the Eucalyptus plant, SEM-EDX was the chosen analytical technique. A suitable digestion procedure was applied to analyze the elemental concentrations of Eucalyptus plant char. The samples were digested with a microwave system with a mixture of nitric acid (HNO_3) and hydrogen peroxide (H_2O_2) [35]. Different elemental concentrations of Ca, P, Sr, Na, K, Ca, Mg, Fe, Mn, Ni, Co, Cr, Cu, Cd, Hg, As, Zn, Se, and Pb were then measured by Inductively Coupled Plasma Mass Spectrometry (ICP-MS, Perkin Elmer NexION350D). All elemental concentrations (CE) were determined using Equation (1), and the instrument was optimized with each element in a standard solution [36].

$$C_E({}^{\mu g}/_{kg}) = \frac{Cs - Cb}{md} * 100 \qquad \ldots\ldots\ldots\ldots\ldots(1)$$

Where C_s= the concentration of metal in the extracted sample (µg/L),
C_b= the concentration of metal in the blank (µg/L),

m_d= mass of the sample (g)

2.2.4. Thermal Analysis of samples

The thermal decomposition of Eucalyptus plant has been studied by the thermagravimetric analysis (TG) and differential thermogravimetric analysis (DTG), in which TG is conducted to obtain weight loss owing to the heating process whereas DTG measures a rate of weight loss with time under controlled temperature conditions. The gasification procedure was performed according to standard conditions at a heating rate of 50°C/min [37].

2.2.5. Proximate analysis

The composition of Eucalyptus plant has been identified in terms of the char samples through the proximate analysis. The ash content, volatile matter, moisture content and fixed carbon were identified for analysis. These conditions were evaluated for the characterization of physical and chemical properties of char samples. The Eucalyptus plant char proximate become useful in characterization of property and predisposition [38].

2.2.5.1. Determination of Moisture

The water content of each plant sample was determined by the method of gravimetric analytical chemistry which measures weight loss by evaporation of H_2O. The water content (%) was calculated by Formula (2) after drying the samples in an oven at 105 °C for 24 h.

$$Moisture\ Content, w\ (\%) = \frac{M_w}{M_D} * 100 \quad \ldots\ldots\ldots\ldots\ldots (2)$$

Where M_w= The mass of water, M_D= The dried mass of the sample.

2.2.5.2. Determination of Ash values

For the present section, a muffle furnace was used, with a 600-650°C temperature over 6 h collectively. The percentage ash content was determined according to equation (3).

$$Ash\ Content, Ash\ (\%) = \frac{Ash_r}{D_{ps}} * 100 \quad \ldots\ldots\ldots\ldots\ldots (3)$$

Where Ash_r= the weight of ash residue, D_{ps}= The dried plant sample.

2.2.5.3. Determination of Volatile matter values

The ash and volatile matter contents of each biomass waste plant sample were determined. Weighing was done after heating the dried samples to 950°C for 7 min and cooling them inside a desiccator for at least 80 min. Equation (4) was then used to calculate the volatile matter (%).

$$Volatile\ matter\ content, w\ (\%) = \frac{Wd - Wf}{Wd} * 100 \ldots\ldots\ldots\ldots\ldots (4)$$

Where W_d= the weight of the dried sample, W_f= The weight of the final sample

2.2.5.4. Determination of Fixed carbon

Fixed carbon (FC) was determined according to the following formulae:

FC (%)= 1-Moisture (%)-Volatile matter (%)-Ash (%)

2.2.6. Ultimate analysis

Ultimate Analysis is an ultimate characterization method for determination of the elementals present in biomass, focusing primarily on major elements; carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) [39]. In the case of Eucalyptus samples found on highways in Iraq, this type of analysis is particularly important to determine what materials are being used. These materials were analyzed using SEM-EDX.

2.2.7. Statistical analysis
All the statistical analyses were conducted using IBM SPP version 26 software. All samples underwent triplicate analyses, resulting in 9 analyses per sample. The results are presented as mean (M), standard deviation (STD), and relative standard deviation (RSD).

3. Results and discussion
3.1. Proximate Analysis of Eucalyptus Samples
The proximate analysis of Eucalyptus plant parts, obtained from Al-Khadra District-Al-Taji-Samarra is shown in the Table 1. The type and level of proximate values were found to be fairly consistent in the three sampling sites. The moisture content varies between 8.108% to 10.452%, and Al-Taji has the highest moisture content. These values are much lower compared to the moisture contents (20-50%) of the biomass being used in thermochemical conversion according to various reports. The ash content is very low, from 3.981 to 4.145%, which is in principle good for energy use since it eliminates the possibility of problems with slagging and fouling. The volatile matter content is very high, from 83.877 to 86.32%; the fixed carbon values were low, from 1.293 to 1.69 %. Thus, from the results obtained, it can be concluded that all the three samples are suitable for the thermochemical conversion processes, namely, for gasification. The energy efficacy level is higher when the ash and moisture content is lower. The low ash content ensures minimal slagging, and moderate volatile matter shows good reactivity for fuel during combustion or gasification. Therefore, Eucalyptus samples from these presented studies depicted better fuel properties due to a lower ash and moisture content compared to the reported studies on other biomass sources such as wheat straw and municipal solid waste, which corresponds to the findings presented by Mierzwa-Hersztek et al., 2019 [40] and Striūgas et al., 2019 [41].

Table 1: Proximate analysis of Eucalyptus plant waste samples from three Iraqi locations

Proximate analysis%				
Sample (Eucalyptus)		Al-Khadra District	Al-Taji	Samarra
Property				
Moist	wt. %(ar)	wt. %(ar)	wt. %(ar)	wt. %(ar)
	Mean value	8.108	10.452	8.68
	Std dev	0.125	0.137	0.123
	%RSD*	1.542	1.32	1.417
Ash	Unit	wt. %(dry)	wt. %(dry)	wt. %(dry)
	Mean value	4.145	3.981	4.053
	Std dev	0.8	0.78	0.45
	%RSD*	19.3	19.59	11.1
V	Unit	wt. %(dry)	wt. %(dry)	wt. %(dry)
	Mean value	86.32	83.877	85.974
	Std dev	0.142	0.132	0.146
	%RSD*	0.165	0.157	0.168
FC	Value	1.427	1.69	1.293

*RSD= (STD/M)×100

3.2. Ultimate Analysis and SEM-EDX Characterization of Eucalyptus Samples
Table 2 below presents the ultimate broken down into CHNOS of the plant samples, as indicated in the Eucalyptus test samples. The values of each parameter are almost the same

across all the three locations. They include carbon, which varies from 32.88% to 33.61%, hydrogen from 5.875% to 5.947%, oxygen from 29.1871% to 31.2405%, nitrogen from 29.4% to 30.62%, and sulfur from 0.5986% to 0.6429%. It is indicative of the results that the samples have the composition of the lignocellulosic biomass and the high carbon and oxygen content, which accounts of its suitability in energy conversion. Figure 1, shows the SEM-EDX results graph. Conversely, all major elements, specifically Al, Si, and Mg, were detected, while heavy metals, including Pb, Ni, and Fe, were traced. The differentiation between CHNOS and SEM-EDX measurements is evident from the fact that while CHNOS gives the general bulk elemental composition, SEM-EDX provides the identification of minor elements and the referencing of their existence. Validating the above statement, Al-Taji SEM-EDX revealed Pb as the most plentiful heavy metal at 19.83%, which was not part of the CHNOS data. However, measuring SEM-EDX is more helpful in environmental risk characterization. The combined use of these techniques provides a holistic understanding of elemental composition and contamination levels, consistent with methodologies in Nzihou and Stanmore [42].

Table 2: The content percentages of C, H, O, N, and S in Eucalyptus plant samples at three locations

Eucalyptus samples	Ultimate analysis in mean (%)				
	C	H	O	N	S
Al-Khadra District	33.61	5.94	29.1871	30.62	0.6429
Al-Taji	32.88	5.875	31.2405	29.4	0.6045
Samarra	33.06	5.947	30.5144	29.88	0.5986

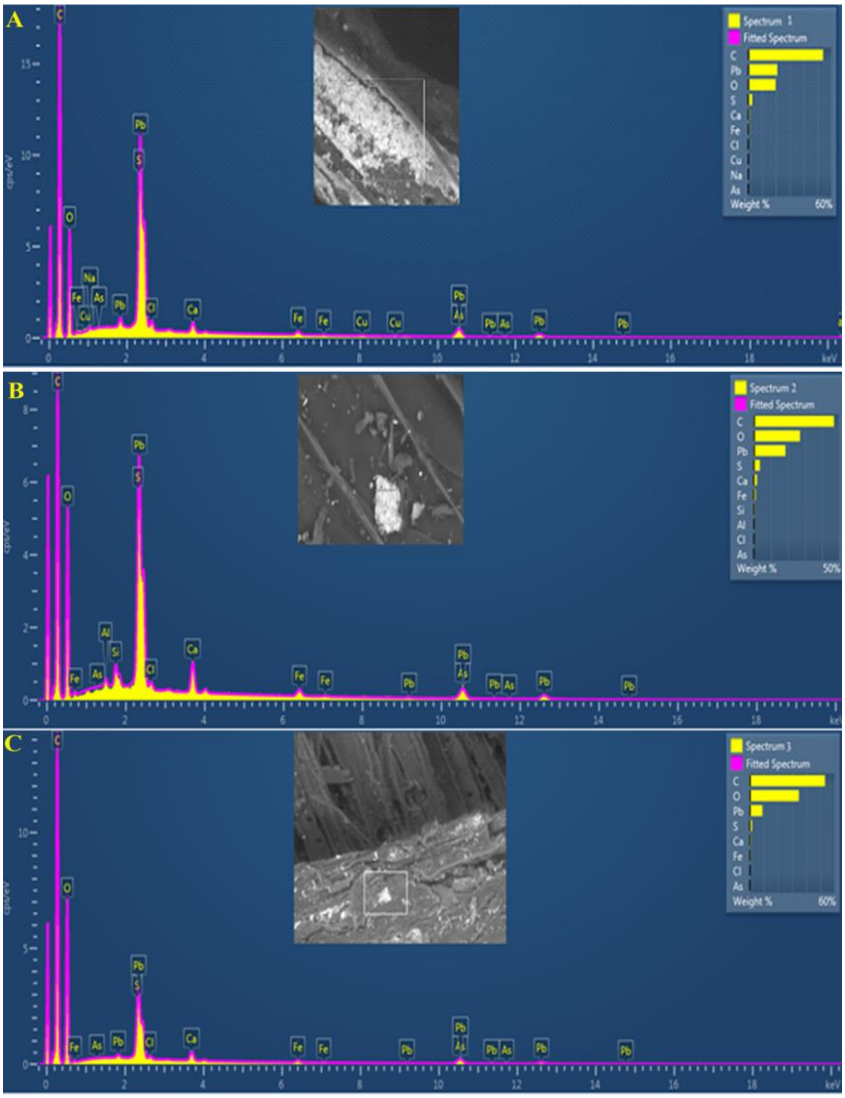

Figure 1: SEM-EDX Elemental Analysis of Eucalyptus Plant Samples from three locations: (A) Al-Khadra District, (B) Al-Taji, and (C) Samarra.

3.3. Distribution of Elements in Eucalyptus Samples by ICP-MS

Figures 2a and 2b summarize the ICP-MS analysis of the major elements (Al, Ca, Cl, K, Mg, Na, Si) and of the minor elements (As, Cu, Fe, Ni, Pb). The samples are from the Eucalyptus tree collected in three different localities. The results showed large differences in both major and minor elements for the different sampling localities. Of the major elements, aluminum shows the highest amount for the samples of Al-Khadra District (1.945%), while magnesium amounts are fairly constant for all localities sampled (0.57-1.095%). Lead shows a very high concentration for the minor elements, which is especially high in Al-Taji (19.825%), which is appreciably above the amounts usually characteristic of varying conditions, and leads to the conclusion that possible contamination in this locality from urban activities should not be dismissed. Arsenic, copper, iron, and nickel in varying concentrations align with findings from Al-Badri et al. (2018) [43] regarding metal distribution in biomass samples. The volatility of heavy metals like Pb and As during thermal processing poses environmental risks, as noted in previous studies by Raheem et al. (2022) [44]. The results underscore the need for site-specific strategies to manage heavy metal contamination during biomass conversion. This study demonstrates how ICP-MS effectively quantifies trace metals, which is integral to assessing environmental impacts during waste-to-energy processes.

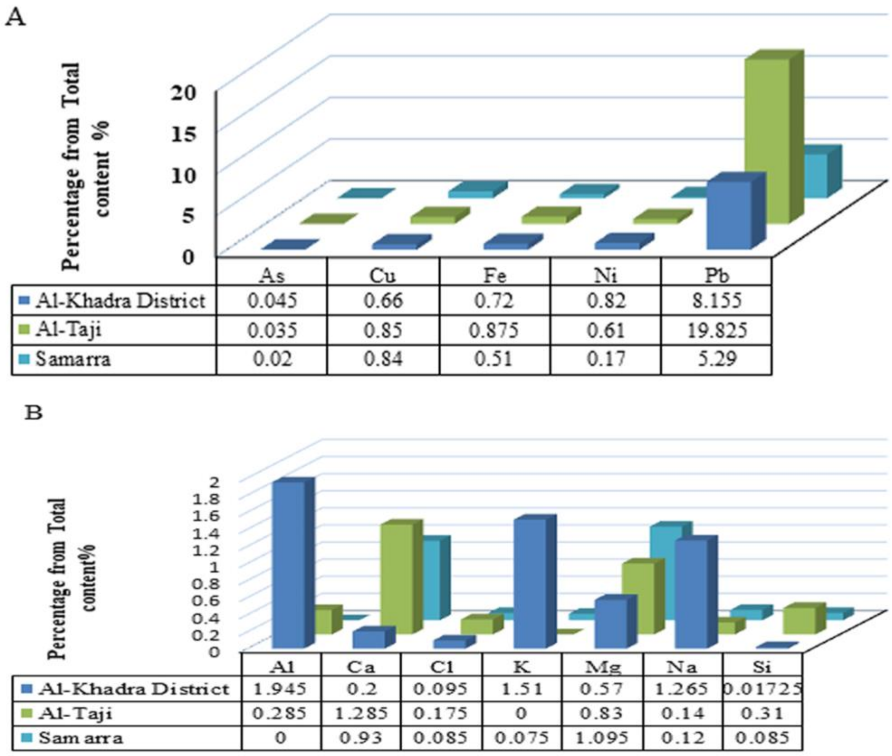

Figure 2: ICP-MS Examination of Principal and Trace Elements in Eucalyptus Plant Specimens. (A): is the major elements (Al, Ca, Cl, K, Mg, Na, and Si) and (B) is the minor elements (As, Cu, Fe, Ni, and Pb) of the Eucalyptus plant from three locations: Al-Khadra District, Al-Taji, and Samarra.

3.4. Thermal Behavior and Metal Distribution in Eucalyptus Gasification Products

Figs. 3 and 4 show the thermal behavior of Eucalyptus samples from the three locations, Table 3 presents Heavy metal concentration in char as produced at various gasification temperatures (700, 800, and 900 °C). The TG curve indicates clear decomposition stages at the temperature region of 0-1000°C, and the weight loss mainly appeared after 200-600 °C with full decomposition approximately to be at 600°C. This pattern is consistent with the literature regarding the normal decomposition of lignocellulosic biomass reported by Fidalgo et al. [45]. The TG and DTG curves (Figures 3 and 4) show a consistent pattern of weight-loss during the thermal decomposition stage, showing a large mass loss between 200-600°C applicable to the decomposition of certain volatile organics and lignin. However, mass losses between the sites can be attributed to the differences in total ash contents and bonding structures of the inorganic compound. It is noteworthy that samples from Al-Taji exhibited the greatest Pb concentrations, which would suggest a greater mass-loss rate. Notably, Al-Taji samples exhibited the highest Pb content, influencing mass-loss rates. The gasification tests at different temperatures (700°C, 800°C, and 900°C) reveal interesting metal behavior patterns.

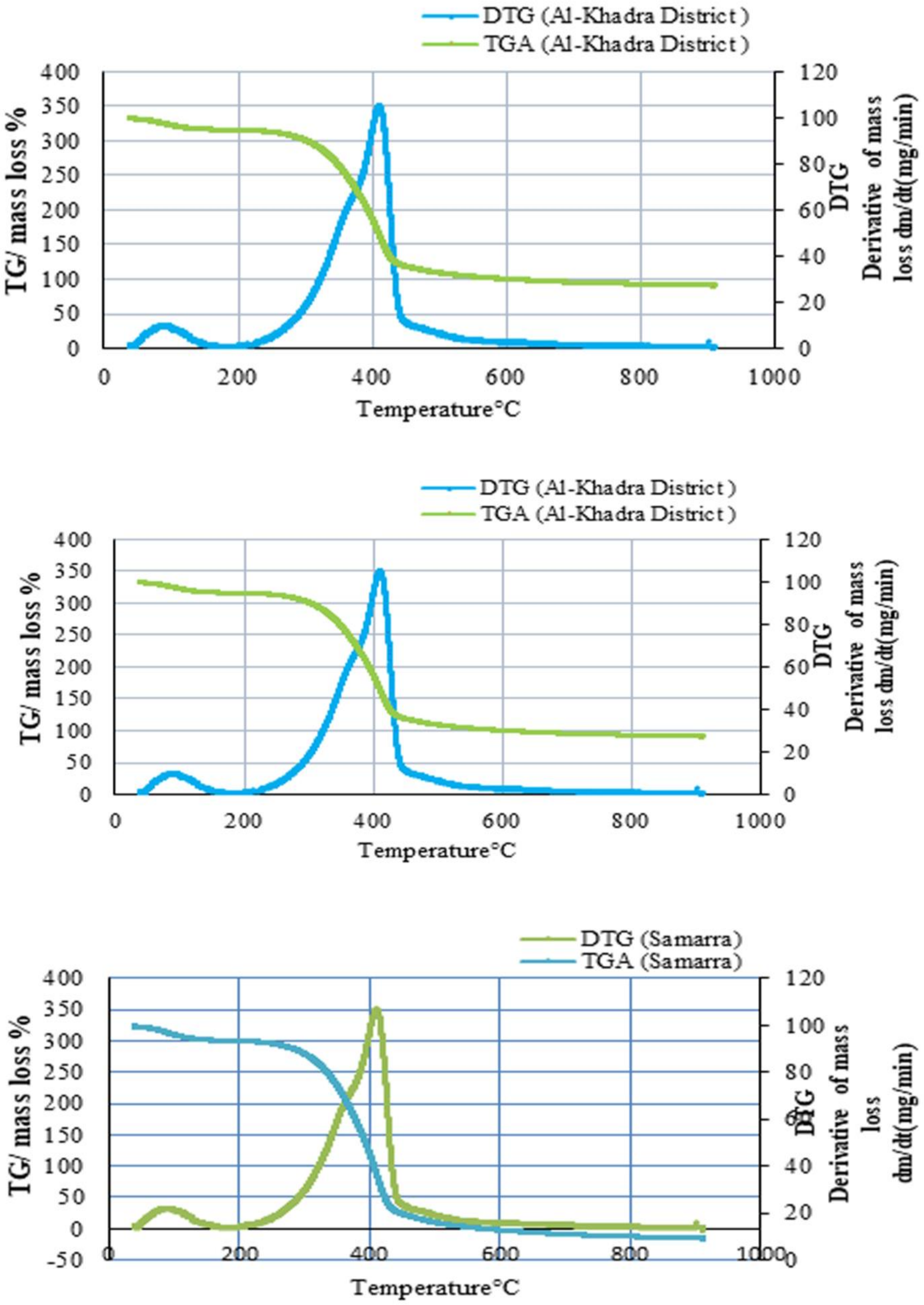

Figure 2: Thermal Behavior and Heavy Metal Volatilization during Gasification of Eucalyptus Biomass

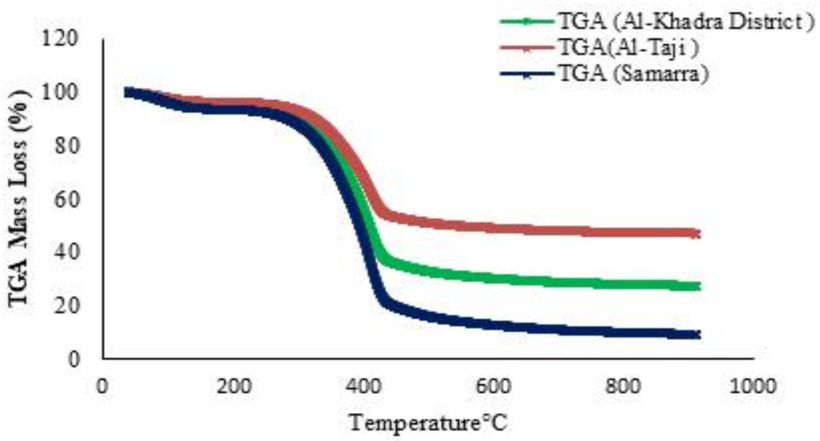

Figure 3. TGA curves Comparison of the thermal behavior of different types of plant waste(Eucalyptus) in gasification under heating rate of 50°C min^{-1}

In Table 3, the gasification behavior of heavy metals is stressed; at 800°C Ni, Cu and Cr metal concentrations increased in char, because the stable solid compounds for As, Ni and Cu result in stable residues; these results are in accordance with works published by Al-Badri et al. 2018. In contrast, the Fe and Na concentrations decreased on increased temperature due to volatilization. The results indicated that out of 800°C optimum terms are attained minimizing the heavy metals discharged but stable char remains, confirming the results of Haynes et al. (2009) [46]. The use of TG/DTG together with ICP-MS has permitted a basic understanding on the thermal behavior and transformations of these metals, which data are necessary in order to optimize gasification processes and alleviate its environmental problems.

Table 3: The concentration of minor elements (Ni, Cu, Fe, As, Pb, Cr, Cd, and Na) of char product from samples (Eucalyptus- Al-Taji) via gasification with temperature (700°C, 800°C and 900°C)

Temperature ˚C	700			800			900		
Metal analysis (mg/kg)	Mean value	Std dev	%RSD*	Mean value	Std dev	%RSD*	Mean value	Std dev	%RSD*
Ni	76.83	0.70	0.91	184.35	106.43	57.74	42.90	0.26	0.62
Cu	244.97	0.35	0.14	755.30	0.61	0.08	201.15	0.17	0.09
Fe	642.67	4.93	0.77	1.01	0.08	7.47	442.37	0.80	0.18
As	2.02	0.11	5.24	39.00	0.26	0.68	0.20	0.04	17.86
Pb	36.77	0.68	1.85	34.83	0.31	0.88	41.70	0.53	1.27
Cr	28.33	1.04	3.67	4.61	0.02	0.33	25.53	0.25	0.99
Cd	2.90	0.01	0.34	457.97	0.12	0.03	1.01	0.01	1.42
Na	686.00	1.00	0.15	184.35	106.43	57.74	338.03	0.15	0.05

*RSD= (STD/M)×100

Conclusion

In an analytical sense this work demonstrated an insight into the transformations and the quantification of heavy metals especially arsenic during the thermal treatment of plant waste. The paper considers the proximate analysis, the ultimate analysis, SEM-EDX, ICP-MS work and thermal analysis as applied to the Eucalyptus plant waste for energy conversion and its environmental implications. It was therefore confirmed that the proximate and ultimate analyses proved exceedingly useful as a low-ash and high-volatile biomass resource. Evidence of significant heavy metallic contamination, especially Pb, in the case of these Al-Taji samples, is presented through SEM-EDX and ICP-MS analysis. Thermal analysis showed optimum gasification within the temperature range of 800 degrees centigrade to preserve an excellent balance between energy recovery and accomplishment of environmental safety. In comprehensive detail, the broad-based integration and adoption of such analytic technologies set up a formidable characterization framework for biomass, tackling the issue of possible heavy metals in thermal conversion but improving the feed quality compared to earlier works related to the subject matter presented here.

Conflict of Interest: The authors confirm that there are no relevant financial or non-financial competing interests to report
Data availability statement: No need
Data deposition: No need
Supplemental online material: No need.

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