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Valorization of Textile Sludge and Cattle Manure Wastes into Fuel Pellets and the Assessment of Their Combustion Characteristics / Gadhi, T. A.; Mahar, R. B.; Qureshi, T. A.; Bawani, M. R.; Khokhar, D. A.; Pinjaro, M. A.; Ansari, I.; Bonelli, B.. - In: ACS OMEGA. - ISSN 2470-1343. - 9:1(2024), pp. 456-463. [10.1021/acsomega.3c05903]

Publisher:
American Chemical Society

Published
DOI:10.1021/acsomega.3c05903

ACS OMEGA

Valorization of Textile Sludge and Cattle Manure Wastes into Fuel Pellets and the Assessment of Their Combustion Characteristics

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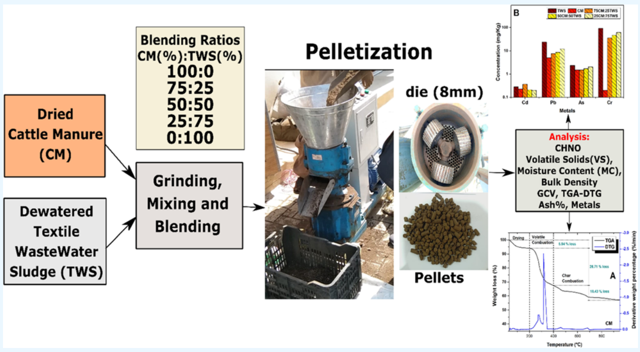
Cite This: *ACS Omega* 2024, 9, 456−463 Read Online



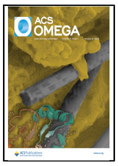
ABSTRACT: The textile wastewater sludge (TWS) treatment and disposal are environmentally challenging due to toxic organics and metals. At the same time, cattle manure (CM), with better combustion performance, i.e., calorific value and uniform burning capability, is still underutilized in many parts of the world. This study evaluated and assessed the TWS and CM blending compatibility to convert them into fuel pellets for the direct combustion option and to stabilize toxic contaminants in TWS. After initial drying, grinding, and particle size control of the raw TWS and CM, both were blended at different ratios. The blended and nonblended TWS and CM samples were converted into pellets and analyzed for proximate and ultimate analyses, namely, moisture content, fixed carbon, CHNO, gross calorific value (GCV), bulk density, ash content, and metals, to evaluate the efficacy for energy applications. Out of three blended ratios, i.e., 75:25 (W/W%; CM/TWS), 50:50, and 25:75, the 75:25 blended pellet composition was found appropriate for fuel application. For the 75:25 blend, the obtained GCV was 12.77 MJ/kg, elemental carbon was 27.5%, volatiles were 41.7%, and residue ash was 42.8% of the total weight. Moreover, the blending ratios of 75:25 and 50:50 revealed that elemental and metal (Fe, Cu, Zn, Ni, Cr, Na, Mg, Mn) concentrations in TWS were stabilized to below threshold limits in the obtained residue ash for safe handling. The explored methods of TWS and CM waste processing, blending, and pelletization proposed a new technique for their sustainable waste valorization into energy sources.

1. INTRODUCTION

The textile wastewater (WW) treatment generates tons of solid organic sludge waste daily, which needs to be disposed of at additional transportation costs from industry to dumping sites. Its disposal is prone to environmental pollution due to its open burning, releases of toxic fumes in the air, and underground leaching. Therefore, exploring methods of properly handling sludge waste and any resource recovery, such as bioenergy, organic fertilizers, chemicals, etc., is important. The generated sludge contains between 2 and 5% by weight of dry organic and inorganic solids[1,2] and varies in amount from 2 to 5 tons per day, i.e., after drying or dewatering, depending upon the types of the textile processes. Because of the dramatic increase in the volume of treated wastewater, a huge amount of sludge is also generated, while its safe handling and disposal occur at additional cost and under environmental regulations. The textile wastewater sludge (TWS) has dominant characteristics of settled entangled cellulose fibers, organic lint, and particulate matter that sediment out during clarifying stages in the effluent treatment plant and is a potential source of bioconversion and resource recovery. Various studies have highlighted that wastewater sludge from the textile industry includes cellulose, various dyes, chemical compounds, and heavy metals. In addition, it is a rich source of organic matter and macro- and micronutrients, including primary nutrients, namely, nitrogen (N), phosphorus (P), and potassium (K), and has significant potential for transformation into biogas and compost.[3−8]

Realizing the rich nutrient and carbon content in TWS, many researchers have investigated various methods, namely, anaerobic digestion, incineration, composting, pyrolysis, cocombustion, advanced oxidation, and blending with concrete. The mentioned methods/treatments were explored to produce renewable biogas, char, compost manure, bricks, fuel materials, and mineralization of their organic components.[9−11] Most of the conducted studies on TWS were on anaerobic digestion alone and/or in combination with other

Received: August 10, 2023
Revised: November 30, 2023
Accepted: December 6, 2023
Published: December 27, 2023



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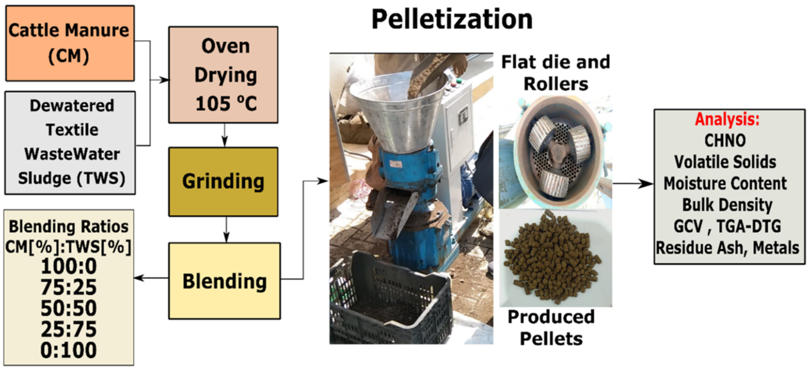

Figure 1. Sequence of processing and conversion of nonblended and blended cattle manure (CM) and textile wastewater sludge (TWS) into pellets and performed analyses; the pelletization machine is by GEMO Company China.

organic substrates, i.e., biogas production and nutrient-rich fertilizers for agriculture.[12,13] However, persistent heavy metals, dyes, and other chemical compounds in TWS are major constraints for its biological treatments, i.e., digestion, composting, and other thermal treatments, i.e., pyrolysis and incineration.[14−17] The direct combustion performance of TWS is weak due to the high content of inorganic materials, and for a similar reason, its heat content is poor. Moreover, studies on combustion/cocombustion performance and dynamics revealed the release of volatile organics in the medium temperature range of 200−300 °C and then unsustained combustion at higher temperature ranges due to significant inert/inorganic content.[9,18] Moreover, the cocombustion of TWS is an area that needs to be further explored, as TWS has a low heat calorific value and high volatile content, which could be optimized by compositing with other organic substrates (potential waste materials) to reduce the problems related to the low combustion efficiency of TWS and also help in solid waste reduction, detoxification, and energy recovery.

Therefore, exploring a more suitable TWS handling and resource recovery method is vital. One interesting aspect could be the cocombustion of TWS, as the direct combustion of TWS is not feasible due to its low heating value.[15,18,19] Some researchers have studied TWS direct combustion or blending with different waste feedstocks, i.e., pomelo peel, bamboo, municipal sewage sludge, and waste tea. They concluded that blending with organic materials or pretreatment improved fuel compatibility, i.e., calorific value, carbon content, volatility, and combustion, and reduced ash content and stabilization of metals.[20]

After transportation, the livestock sector is the biggest greenhouse gas emitter, and cattle waste/manure (CM) is the major contributor to different livestock sources. The openly dumped and decayed CM emits CH_4 and CO_2; only a small portion is used for composting or farmyard manure application. Much of the research has revealed the renewable energy potential of CM through anaerobic digestion or direct combustion.[21,22] Szymajda et al.[23] reported the successful conversion of CM into solid fuel pellets for easier compression and use as sustainable mono-/cocombustion fuel. Few researchers[19,24] have assessed the TWS properties for cocombustion options by studying the behaviors of ash formation, thermogravimetric decomposition, and formation of different oxides in residue ash.[19,25] The same study concluded that the 1:1 ratio of TWS/CM is suitable for thermal reactions. However, other critical fuel characteristics were not considered in the mentioned study, i.e., GCV, the fate of heavy metals, and conversion into fuels. Still, some research gaps lie in the TWS and CM transformation into a suitable form of bioenergy, detailed analyses of energy performance, and the fate of toxic contaminants. For the first time, the current study revealed the methods and procedures of processing (drying, grinding, particle size control) the raw CM and TWS, their blending into different weight ratios, and pelletization to convert them into fuel pellets, along with a detailed analysis of the fuel characteristics and metals of the produced pellets. The CM and TWS samples were blended in different ratios after optimized drying and grinding and particle size controlling steps to produce bulkier pellets and were analyzed through GCV, thermogravimetric analysis (TGA)-derivative thermogravimetric (DTG), bulk density, and metals to explore the best blending ratio of CM and TWS compatible for direct combustion and metal stabilization.

2. MATERIALS AND METHODS

2.1. Materials. The seven-day-old CM was collected from the dairy farm, and the dewatered TWS was collected from Al-Rahim Textile Industries Pvt., Ltd., Pakistan. The analyzed moisture contents of as-received CM and TWS were around 58 and 52%, respectively. Before blending different ratios, the as-received CM and TWS were dried overnight in an oven dryer at 105 °C to obtain the moisture content to 10% for easier grinding. Later, the dried CM and TWS were separately ground in the grinding mill for 3 min at around 10 000 rpm to obtain the fine powder, which was further sieved in a 5-gauge mesh to obtain the homogeneous size $\leq$ 4 mm, which was found effective for the pelletization process. Around 60 kg of final weight (after drying, grinding, and mesh sieving) of each CM and TWS were prepared for repeatable blending and pelletization.

2.2. Blending and Pellet Conversion. For reproducible results, two different batches of dried, ground, and sieved CM and TWS with particle sizes of $\leq$4 mm were blended in a ribbon mixer blender at 75:25, 50:50, and 25:75% ratios (CM/TWS, weight on a weight basis) for 15 min blending. Each batch of blended and nonblended CM and TWS was around 10 kg. The blended and nonblended batches were separately converted into pellets using a flat D-type pelletization machine (by GEMCO company, China) with a configuration of a moving die disc during pellet production. This machine was configured with a die disc at groove sizes of 8 mm (pellet diameter) and produced pellet cutter settings at 244 mm in length, as shown in the digital image in Figure S1 in the Supporting information. The pelletization machine compresses

Table 1. Proximate and Ultimate Analyses of Blended and Nonblended CM and TWS

analysis	CM (100)	TWS (100)	CM/TWS (75:25)	50:50	25:75	previous studies (CM; TWS)[2,23,31,32]
moisture [%]	4.8	13.5	7.3	8.1	9.8	4.36−8.66; 5.02
volatile [%]	43.6	40.4	41.7	42.4	42.6	52.8−57.86; 34.72−54.52
ash [%]	39.3	43.4	42.6	43.5	44	22−31.16; 39.32−60.26
fixed carbon [%]	12.3	2.7	8.7	5.4	3.8	11.41−11.68; 0.96−6.15
C [%]	30.5	17.4	27.3	25.3	20.1	32.25−37.6; 16.23−24.67
N [%]	1.6	1.4	1.6	1.6	1.2	1.63−1.84; 0.96−3.1
H [%]	4.1	4.6	4.5	4	4.3	5.26−5.69; 2.51−5.10
O [%]	35.87	25.65	33.80	31.19	29.31	24.51−35.44; 11.82−25.9
bulk density [kg/m^3]	594	663	632	641	646	
GCV [MJ/kg]	15.58	8.64	12.77	11.64	10.67	13.41−15.86; 4.11−10.17
GCV [kcal/kg]	3723	2074	3065	2793	2561	

the powder material into bulkier pellets with different diameters, lengths, and bulk densities. The produced pellet composition depends on the groove size of the pelletizer, the cutter setting, and the moisture of the material for intended applications, i.e., energy, fodder, and fertilizers. The pellets could vary in diameter and length sizes, composition and moisture ratios depending on the fed raw materials, disc die grooves, and cutter sizes. For comparison, the nonblended CM and TWS were converted into pellets, i.e., following the same procedure. The digital images of the pelletization process and produced pellets are shown in Figures S1 and S2 in the Supporting information. The scheme in Figure 1 shows the processes of converting CM and TWS into pellets and performed analyses to evaluate the composition and properties of obtained pellets.

2.3. Characterization and Analytical Methods.

Five samples of each batch of blended and nonblended pellets, i.e., nonblended CM and TWS and three blended ratios, were analyzed through proximate and ultimate analyses. The proximate analysis gives the gross composition of as-received fuel/biomass, i.e., ash content, moisture content, volatile matter, and fixed carbon. The ultimate analysis measures the weight % of C, H, N, and O, where oxygen is commonly estimated by difference.[26]

The blended and nonblended pellet samples were ground to below 250 μm particle size. The proximate analysis was performed using a LECO TGA701 following the ASTM D7582-15 standard procedure. The ultimate analysis was performed on an ASTM D5373-compliant LECO CHNO Truspec microanalyzer. The GCV values were calculated using the empirical equation (eq 1), reported by Mesroghli et al.[27]

$$\mathrm{GCV}\left(\frac{MJ}{Kg}\right) = 37.777 - 0.647M - 0.387A - 0.089\mathrm{VM}\quad R^2 = 0.97 \tag{1}$$

where M is the moisture content, A is the ash content, and VM is the volatile matter.

TGA was performed in an STD Q600 (micro TGA) per the prescribed technique.[28] The temperature and sensitivity of the equipment were calibrated by using phase transition events for the samples. The samples were heated at 10 °C/min from 30 to 950 °C. The sample weighed 15 ± 0.1 mg. The flow rate of the air was set to 50 mL/min. Each sample was examined at least twice to confirm the repeatability of the results. The DTG curve is obtained by taking a weight loss curve derivative to compute the change rate in mass. The baseline was adjusted and normalized using Origin 9.0 software.

The wet digestion method was followed for the metal analysis of the residue ash of blended and nonblended pellets, i.e., collected ash after the muffle furnace combustion at 700 °C for 1 h duration. 1 g of the residue ash of each pellet sample was ground and screened through a 100-mesh sieve. The ground sample was placed in a crucible, the first 10 mL of concentrated nitric acid was added, and the crucible was placed on a hot plate and slowly heated at 130 °C.[29] The sample volume was continuously monitored, and when evaporated to about 2 mL further, 5 mL of nitric acid was added and heated at 180 °C until the fumes tuned into the white, transparent color of the resultant solution. The final prepared sample was transferred into the flask, and deionized water was added up to 25 mL for dilution. The digested sample was filtered through a 0.45 μm filter and stored in a refrigerator for analysis. The inductively coupled plasma mass spectrometer (ICPMS) NexION 350 by PerkinElmer (USA) was used to quantify the elemental analysis (including heavy metals) of the samples. During the ICPMS analysis, the analyte sample is fragmented into ions/atoms to detect any element's total elemental mass independent of its valence or chemical state. For the reproducibility of the results, the metal analysis was done for combusted pellets of two batches, and average concentrations of each metal were considered to report.

The loose bulk density of the obtained pellets was measured following the reported method.[30] In brief, the pellets were filled in the known volume of the cylinder and weighed before and after filling. The bulk density is measured through the relationship (weight of filled cylinder (kg) − the weight of empty cylinder (kg))/cylinder volume (m^3). The obtained values of the bulk densities of two batches of each pellet sample were averaged to report.

3. RESULTS AND DISCUSSION

3.1. Proximate and Ultimate Analyses.

Table 1 shows the proximate and ultimate analyses of nonblended and blended CM and TWS, along with the calculated bulk density and GCV values, compared to previously reported results. The obtained carbon content in CM was higher than the TWS, due to which the GCV value of CM is higher, i.e., 15.58 MJ/kg, than the 8.64 MJ/kg value of the TWS (calculated using eq 1). Previous studies also report similar trends.[18,23,31] The obtained residue ash content of CM is slightly lower, i.e., 39.3%, than 43.4% of TWS, but comparatively higher than that reported previously.[18] The residue ash content depends on various factors, i.e., types of cattle feed and the presence of foreign and inorganic residue mixed with the CM. The loose bulk density of the CM was lower than that of the TWS. In the case of CM

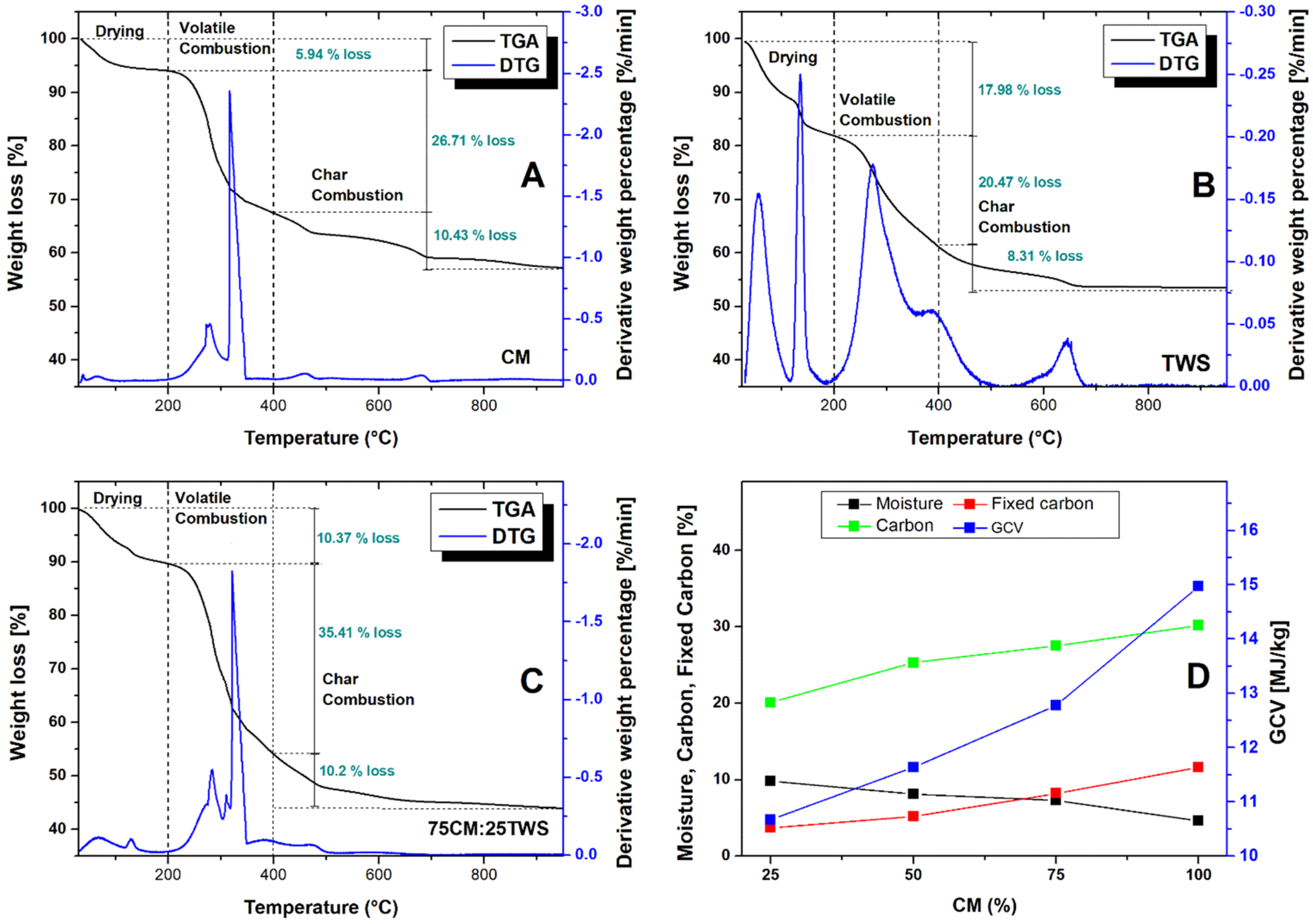

Figure 2. TGA-DTG curves of (A) CM pellets, (B) TWS pellets, (C) blended ratio of 75:25, and (D) changes in moisture, carbon, fixed carbon %, and GCV at the increased/decreased CM ratio.

pellets, the obtained bulk density was around 594 kg/m^3, higher than that reported earlier, i.e., 471.4 kg/m^3.[23] The improved bulk density in CM could be due to its fine grinding and size homogenization for the pelletization process. The TWS pellets have a loose bulk density of around 663 kg/m^3, meeting the standard levels of ≥600 kg/m^3 (EN ISO 17225-2 A1, ISO 2014). The difference in bulk density is associated with the finer particles of sludge that allowed a high compression and bulkiness compared to the loose aggregates of CM. The lower value of CM's bulk density than that of TWS revealed CM's lower moisture content behavior, which could be due to voids and gaps in CM pellets that allowed better drying than the bulkier TWS pellets.

In the case of blended samples, the change in values was correlated to the properties of CM and TWS alone. For the CM/TWS (75:25) ratio, a slight decrease in fixed carbon and elemental C was observed, i.e., 8.7% fixed carbon and 27.3% elemental C, compared to 12.3 and 30.5% in the case of pure CM. The resultant GCV value of the 75:25 blend was around 12.77 MJ/kg, with 42.6% residue ash, and the loose bulk density was around 632 kg/m^3, improved compared to pellets of CM alone. However, the other two blended ratios of 50:50 and 25:75, at increased TWS content, resulted in poor fuel compatibility with low GCV values of 11.64 and 10.67 MJ/kg, low fixed carbon of 5.4 and 3.8%, and high amounts of ash of 43.5 and 44%, respectively. Only the bulk density of 50:50 and 25:75 ratios was slightly higher, i.e., 641 and 646 kg/m^3, respectively, than the 75:25 blend due to a higher ratio of TWS.

3.2. TGA.

Figure 2A–C shows the TGA-DTG curves of the pellets produced from CM and TWS alone and a blended ratio of 75:25 (CM/TWS). In the case of CM pellet monocombustion, the TGA-DTG plot (Figure 2A) shows the loss of moisture until 200 °C, and after an increase in the temperature gradient to 225–250 °C, the CM fragmentation starts with a high and continuous loss of volatile matter in the temperature range of 300–350 °C. During 400–700 °C, the loss of residue carbon occurs. Some authors[18,24] categorized these changes into three stages: (1) the drying stage at 30–200 °C, mostly associated with loss of water; (2) the volatile combustion stage at 200–400 °C, where CO_2 and CO are released in high quantities, originating from the loss of elemental carbon and combustion reactions with the release of alcohols, ethers, ketones, aldehydes, and acids;[24] and (3) the char combustion stage, where mostly fixed carbon is lost along with loss of nitrogenous compounds.

In the case of TWS pellet monocombustion, the TGA-DTG curve (Figure 2B) showed a significant loss of 20% during the drying stage, which suggests that along with the loss of water, some less-stable volatile dyes and polymers were volatilized with the subsequent loss of volatile matter and residue carbons during the volatile and char combustion stages. In the case of the blended sample, i.e., 75:25 (CM/TWS), the TGA-DTG (Figure 2C) shows the correlated behavior of weight loss and

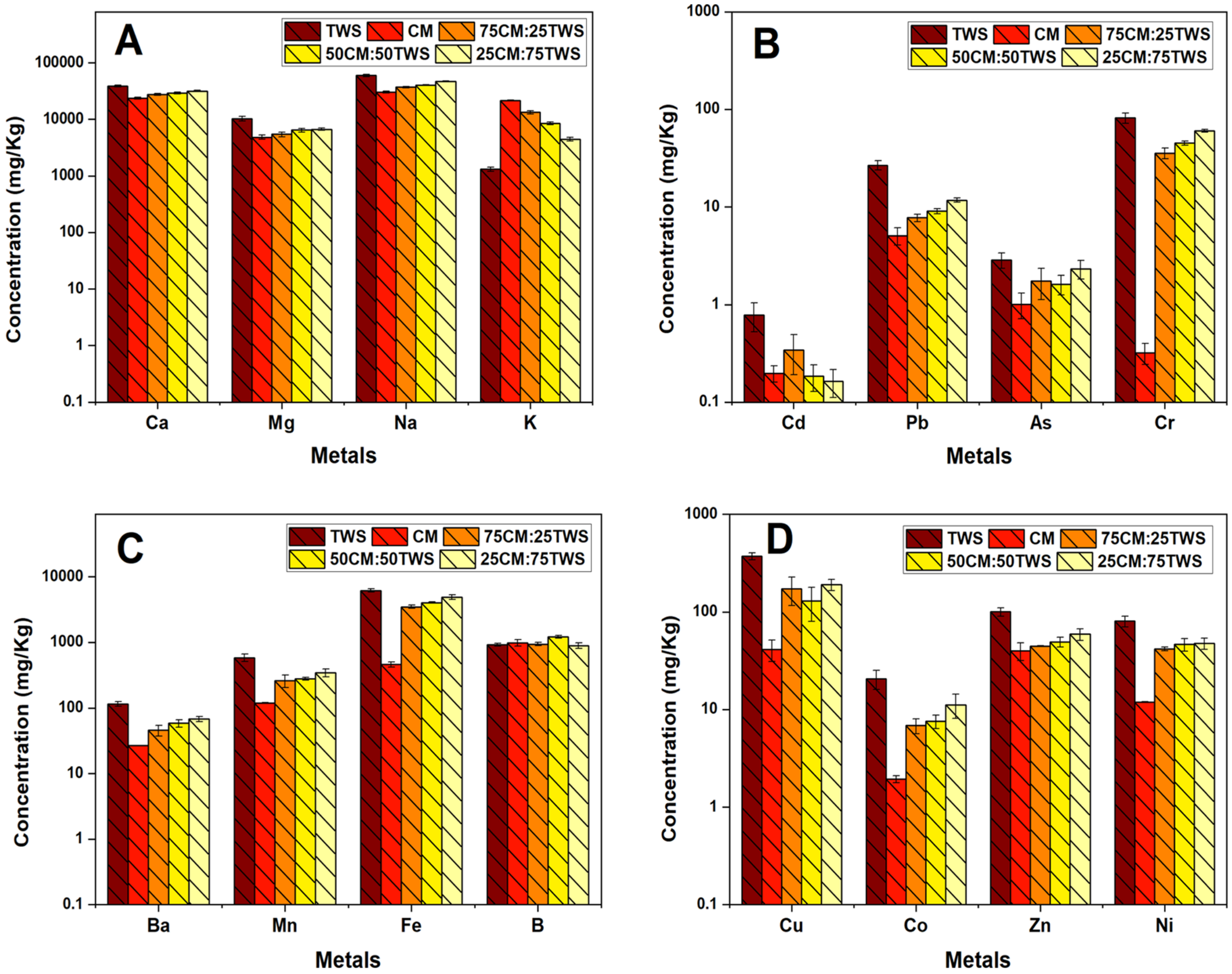

Figure 3. (A−D) Average concentration of different metals found in the residue ash of combusted CM and TWS pellets and their blends in the log scale.

fragmentation compared to CM (Figure 2A). However, the trend of weight loss in the volatile combustion stage is greatly affected due to the presence of TWS because during 200−400 °C, the CM monocombustion lost about 35% of the weight, while the CM/TWS blend cocombustion resulted in a weight loss of 46%. Overall, the TGA-DTG results revealed that organics in the TWS are highly volatile and less stable in drying and volatile combustion stages. Therefore, its compatibility for cocombustion should be adjusted at low ratios to maintain the combustion characteristics.

The information from the ultimate, proximate, and TGA-DTG analyses data is plotted in Figure 2D, showing the changes in moisture content, elemental and fixed carbon, and GCV of the CM and TWS blended and nonblended pellet samples. Adding TWS to CM or vice versa significantly impacts elemental and fixed carbon and GCV values due to high volatility and ash residues associated with TWS. Similarly, in correlation to TWS, adding high ratios of CM improves the fixed carbon, elemental carbon, and GCV values and slightly reduces moisture and ash content. Similar trends are reported by other studies, where TWS cocombustion characteristics are reportedly improved after adding organic feedstock, i.e., livestock waste, pomelo peel, bamboo, municipal sewage sludge, and waste tea.[10,18,24,32]

3.3. Analysis of Metals.

One of the critical concerns of TWS treatment and handling is to manage the fate of heavy metals because the presence of Cr, Ni, Zn, Fe, Al, and other elements, i.e., Na and Ca, above the threshold/standard limits are reported.[14,15,33,34] The presence of metals in high concentrations is critical from microbial digestion, incineration, and monocombustion treatment methods, as these methods are associated with the loss of organic and volatile content and fixing of inorganic elements and metals in the residue ash or resultant final material. The metal concentration of the raw fuel material and residue ash varies due to the loss of organics and volatiles in the energy fuel before and after combustion.[35,36] Figure 3A−D shows the concentrations of different metals in the pellet residue ash after combustion and loss of carbon and other volatiles. In the residue ash of the TWS nonblended sample, the highest concentration of Na, i.e., 59 474 mg/kg (around 6%), was observed, while in the case of CM alone, it was around 31 050 mg/kg (3%). The blended ratios suggested a slight decrease in Na concentration, i.e., 37 150 mg/kg. After Na, the concentrations of Ca were in higher ranges, i.e., up to 40 253, 22 947, and 27 484 mg/kg for nonblended TWS, CM, and blended 75:25 ratio, respectively. The concentrations of Mg and Fe were also on the higher side in TWS alone (1 and 0.65%, respectively) compared to CM alone, which were adjusted in the blended samples. The concentration of other heavy metals, i.e., Cu, Cr, Zn, Ni, and Ba, were found on the higher side in the TWS sample, i.e., 404, 92, 90, 91, and 106 mg/kg, respectively, while the B concentration was found in the same range of 975−990 mg/kg for both CM and TWS, and Mn was higher in case of TWS. Cd, Pb, and As concentrations were found in the lower ranges, i.e., 1−10 mg/kg. Some researchers[15,18] reported a high aluminum (Al) concentration in TWS due to the alum-based coagulation in the primary textile effluent treatment. However, for this study, Al was not analyzed.

Interestingly, the concentration of the majority of the elements was significantly reduced in the blended samples. Table S1 in the Supporting information shows the values of the elemental concentrations in the analyzed TWS and CM blended and nonblended samples compared to the standard threshold limits of some heavy metals.[15] The blended sample 75:25 (CM/TWS) showed a stabilized concentration of heavy metals compared to the standard threshold standards in their total elemental form only and not specific to any valence, chemical, or ionic state.

4. CONCLUSIONS

This study was designed to utilize TWS and CM wastes to produce sustainable energy alternatives. The initial processes of drying, grinding, particle size control, and optimized blending of raw CM and TWS were optimized to produce fine quality bulkier energy pellets with an average diameter of 8 mm and a length of 244 mm. The produced pellets from nonblended and blended TWS and CM were assessed to evaluate their potential for mono-/cocombustion and fuel application efficacy. The produced five samples of pellets, i.e., CM (100), TWS (100), and CM/TWS (75:25, 50:50, and 25:75) in two batches, were evaluated through proximate and ultimate analyses, and later, the combusted pellets were analyzed for the concentrations of metals. The proximate and ultimate analyses revealed the CM potential for monocombustion with a GCV of around 15.58 MJ/kg, while the nonblended TWS pellets have high weight loss, less volatile stability, low GCV, and high ash residue. The pellets with the CM/TWS blend ratio of 75:25 were suitable for direct combustion with a compromised GCV of 12.77 MJ/kg, an ash residue of 41%, and a bulk density of 624 kg/m^3.

Moreover, the concentrations of metals, i.e., Na, Fe, Cu, Zn, Cr, Ni, B, and Mn, were also found on the higher side in the ash residues of the combusted TWS pellets. However, a significant decrease and stability in metal concentration were observed for the CM/TWS (75:25) blend compared to the standard threshold limits. Overall findings revealed the conversion and value-addition potential of CM and TWS and their blends into transportable and directly combustible fuel pellets for energy applications and utilization of abundant waste biomass as alternative fuels. The outcomes of this study could be important for textile industries' energy needs with proper handling, treatment, and resource recovery of TWS. The explored methods and outcomes on CM valorization into combustible and transportable energy pellets could be viable for household and industrial energy needs. Furthermore, the current study revealed research avenues of the valorization compatibility of TWS and CM bioenergy with other waste biomass for improved results.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.3c05903.

Concentration of heavy metals with their threshold limits and images of feedstock blending (Table S1, Figures S1 and S2) (PDF)

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Author Contributions

T.A.G.: conceptualization, methodology, investigation, formal analysis, and writing—original draft. T.A.Q.: conceptualization, methodology, investigation, formal analysis, and writing—original draft. R.B.M.: supervision, project administration, and funding acquisition. M.R.B.: formal analysis, validation, and writing—review and editing. M.A.P.: formal analysis and writing—review and editing. D.A.K.: formal analysis and writing—review editing. I.A.: sample characterization and analysis. B.B.: result validation and writing—review and editing.

Funding

This study was funded by the Higher Education Commission (HEC), Pakistan, under the research project Technology Development Fund (TDF) HEC-TDF-03-037.

Notes

The authors declare no competing financial interest.

Statement of Novelty For the first time, the current study optimized the methods and procedures of drying, grinding, particle size control, and blending of cattle manure and textile sludge waste biomass to valorize them into bulkier fuel pellets, along with a detailed analysis of the produced pellets' fuel characteristics and toxicity stabilization. The previous work mostly focused on anaerobic digestion and incineration techniques. Overall findings revealed the valorization potential of CM and TWS and their blends into transportable and directly combustible fuel pellets for energy applications.

■ ACKNOWLEDGMENTS

The authors acknowledge the technical support of Dr. Fahad Irfan Siddiqui, Department of Mining Engineering, Mehran UET Jamshoro, Pakistan, and Najeebullah Channa, DISAT, Politecnico di Torino, and the administrative support of the executives of Al-Rahim Textile Industries, Pakistan.

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