PROGRESS TOWARDS THE STANDARDIZATION OF TEST PARAMETERS AND PROTOCOLS FOR EVALUATING DENSIFIED FUEL PRODUCTS.

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Densification technologies used in managing biomass and coal fines to produce fuel briquettes or pellets are gaining rapid acceptance and application due to the widespread uptake of renewable energy. This has resulted in the mushrooming of many standard and non-standard evaluation tests for densified solid fuels in this industry. These tests are sometimes inconsistently applied especially if the available options for evaluation, underlying assumptions and principles of the test methods chosen are misunderstood. A critical review of the fuel briquettes evaluation tests to understand their relevance, strengths and limitations is necessary to advance public and research knowledge. This study investigated current methods of standardization to identify best practices, inconsistencies, trends and gaps in the application of fuel briquettes testing protocols. Procedures for evaluating combustion or thermal properties include calorific value, burn rate, open air combustion test and water boiling test; ignition time; after-glow test; specific fuel combustion or thermal efficiency were discussed. Physical and chemical property evaluations involved densities and related ratios, proximate and ultimate analyses. Equally important are mechanical parameters frequently informed by tensile/compressive strength, impact resistance, friability and water resistance. As renewable densified fuels gain more global popularity, it is critical to have a uniform standard of evaluating their quality.

Briquette, densification, biomass, solid biofuel, evaluation methods, analytical techniques

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1. Introduction

The depletion, price instability, and polluting effects of fossil fuels versus the increasing need for energy security and cleaner fuels that offer climate change mitigation benefits continue to spur technological advancements in the fuel industry. Because they are abundant in nature and carbon neutral, lignocellulosic biomass feedstocks, such as energy crops and agricultural and forest residues, present a potential replacement for fossil fuels. However, the low bulk density of lignocellulosic biomass must be addressed in order for it to be used as a biofuel, either directly or indirectly. One effective way to get around this issue is to densify biomass into cubes, briquettes, or pellets, which have advantages over heterogeneous feedstocks, including uniform shape and quality. Such consistent properties make densified fuels easier to use in available conventional infrastructure for thermochemical conversions that include pyrolysis, gasification and combustion, as well as biological conversions [1]. Besides facilitating the increased use of biomass in the fuel industry, densification technologies are also an increasingly acceptable route for managing solid fuel fines which when left unattended contribute to air pollution [2]. Generally, densification involves the mixing of solid fuel fines with or without a binder. Thereafter, pressure is applied to this mixture in a mould, through a screw, hydraulic or mechanical press. Briquetting without a binder requires higher temperatures and pressures since the product obtains its binding properties from inherent natural binders within biomass, such as lignin. After being subjected to pressure, solid fuel particles stick to each other and form a denser composite particle called a briquette or pellet which has better mechanical handling and thermal properties than the original undensified powder. The technology has been intensely studied and applied to sawdust [3], coal fines [4], and other biomass residues [5], [6]. Densification of mixed materials (co-briquetting or co-pelletization) has also been successfully performed [7], [8]. Based on a cursory examination of the literature on Google Scholar for the period from 2008-2022 (Fig 1), there is evidence of a consistent growth in research on biomass densification [1].

At commercial scale, wood pellet production has increased steadily worldwide between 2005 and 2019 reaching over 40 Mt, and is predicted to surpass 60 Mt by 2025, highlighting the significance of densified biomass products in renewable energy systems [1]. Moreover, more diversified densified products have been witnessed during these periods, with variations coming from the feedstock (raw straw or woody biomass; torrefied and carbonised feedstocks) and machines used.

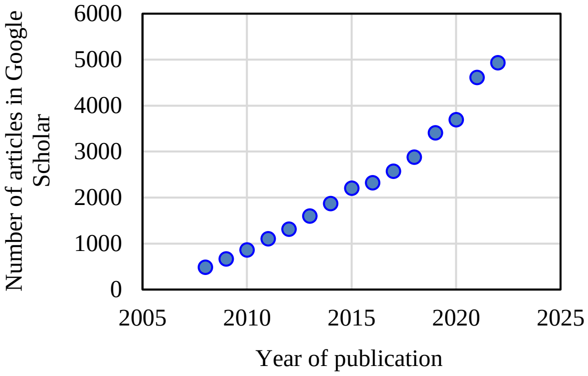

Fig 1 Published papers on biomass densification from 2008-2022

Only certain developed regions that have explored bioenergy usage at large scale, such as Europe, Australia and China, have developed a few measurement standards for size, dimensions, density and calorific value, while some of the densified fuel quality parameters such as combustibility and impact resistance have widely varying test protocols [9]. As the densified fuels industry expands, the subject of product quality, uniformity, and standardization of testing protocols becomes more pertinent.

Good quality densified fuels should have mechanical strength to resist fracture during transportation and storage. Briquettes should also have better combustion characteristics than the initial undensified materials. These mechanical and combustion characteristics are evaluated using various parameters that are derived from standard and non-standard tests and techniques. Some of these tests are only applicable to certain situations, for example, lignocellulosic compositional analysis on biomass raw material cannot be performed on coal [10]. Some briquette tests focusing on mechanical properties are more applicable to high pressure commercially processed briquettes only and not for low pressure, low-tech artisanal products [11]. Other researchers think the briquette application rather is what should govern the required mechanical properties measurement, not the process. For example, briquettes meant for industrial fuel purposes, are expected to have better integrity and durability considering storage and transport as opposed to those destined for domestic cooking where less handling and short storage times apply [12]. Additionally, some briquette tests convey the same information about the performance of the briquette for example the "burn rate'' and the "flame propagation". Both parameters give an indication of how long the briquettes will last burning under certain conditions. Therefore, a one-size-fits-all approach to briquettes characterization protocols is not possible although there is scope and a clarion call for standardization of some of these tests [12]. Standardization of tests would benefit commercial producers who would want to tap into the lucrative export markets.

Several researchers include in their article titles phrases such as briquette tests, briquette evaluation, briquette characterization, briquette analysis, but they report only on parameters of their choice, which are often different from parameter sets selected by the other researchers. A few publications are selected in Table 1 to show the variations in type of briquette analytical parameters presented in literature by various researchers.

Table 1 Sets of briquette analytical parameters reported in some selected published articles

Reference	Briquette parameters													
	Moisture content	Proximate	Ash content	Calorific Value	CHNS	Density	Impact resistance	Friability index	Water resistance	Ignition time	Burn rate	After glow time	Smoke time	Internal bonds
[13]	✓	✓	✓	✓	×	×	×	×	×	×	×	×	✓	×
[14]	×	×	×	×	×	×	✓	×	✓	×	×	×	×	✓
[15]	✓	✓	✓	✓	×	✓	✓	×	×	✓	✓	×	×	×
[5]	✓	✓	✓	✓	✓	✓	✓	✓	✓	×	×	×	×	×
[16]	✓	✓	✓	✓	×	✓	✓	×	×	✓	✓	×	×	✓
[11]	✓	✓	✓	✓	×	✓	×	×	×	✓	✓	×	×	✓
[10]	✓	✓	×	✓	✓	×	×	×	×	×	×	×	×	✓
[9]	✓	✓	✓	✓	×	×	×	×	×	×	×	×	×	×
[17]	✓	✓	✓	✓	×	×	✓	✓	✓	✓	×	×	×	×

✓ Parameter is reported in the article;
× Parameter not reported in the specific article

As the sample in Table 1 demonstrates, there is no consensus among researchers in terms of what constitutes a standard full characterization set of parameters for densified fuels as identified from information in literature. Ward, Yacob and Montoya [18] characterized briquettes and reported mechanical properties only but did not perform other common combustion tests that yield parameters such as burn rate, glow time, and combustion efficiency. Trubetskaya et al. [10] used expensive spectroscopic instruments to report detailed morphology and internal bonding briquette parameters but their study did not address any of the briquette mechanical and cheap, easy to perform combustion parameters. Although their article addressed a knowledge gap, the authors of this review article opine that research investigating densification should always cover mechanical and combustion parameters, which should be the main motivation behind fines densification for fuel production. If mechanical and combustion properties had been incorporated in the previous study mentioned, it would then have assisted in mapping how the morphology and internal bonding parameters were related to pertinent mechanical and thermal parameters. Moki et al., [19] performed and reported a less common briquette parameter called flame propagation. This parameter indicates how fast a flame traverses along the length of a solid fuel from one end to the other and this may possibly be easily correlated to burn rate. Many researchers rarely report this parameter perhaps because it is not easy to measure for spherical or small (lengthwise) briquettes.

The pollution potential of briquettes when burned was tested and reported by Pandey and Dhakal [20] by evaluating the smoking time and particulates content. Limited publications focused on evaluating the aspects of briquettes pollution potential. Several review and research articles have been published on the briquetting technology focusing on different aspects such as binders' type and concentration [21], [22], effects of co-briquetting [15], substrate particle size effects [23] and at times a combination of these aspects [24], [25], [26]. However, there are very limited works in literature that focused on an in-depth analysis of the issues pertaining to analysis and evaluation of briquettes as a standalone subject. Dohm et al. [28] made a good attempt to evaluate the methods used to characterize the durability of coal-biomass briquettes, specifically covering compressive strength, impact-shatter resistance and abrasive/friability resistance. They also recognised that, though researchers agreed on important briquette properties to be measured, specific test methods used to determine these parameters are 'highly variable'. They used statistical analyses comprising checking normality of distribution, scatter of data, standard deviation and confidence intervals to check on the reliability and replicability of the various tests. Their evaluations were, however, not exhaustive as they did not cover combustion parameters and excluded some mechanical test protocols. The inconsistencies in briquette quality tests make it difficult to compare the performance of various technologies and formulations and impedes the quick adoption of such products by industry and market. The study advances knowledge by highlighting where there are standard tests and the frequency of adoption for such procedures. It also pinpoints gaps and inconsistencies in current briquette evaluation techniques in a bid to trigger dialogue for the development of standard tests from some of the frequently used briquette analytical methods in this fast-developing industry.

The review mostly covered recent articles within the last 15 years (2008-2022) to obtain a more comprehensive trend analysis of the briquette evaluation techniques and the associated inconsistencies. The interest was in original research articles, where authors conducted tests on briquettes and an excess of seventy such articles have been discussed and analysed in the review. Review articles were only discussed in the process of justifying the need for a review such as this. Google, Google Scholar and Scopus search engines and journal search sites such as Science Direct were explored using the keywords presented in this article, and their relevant combinations.

2. Standard parameters and common analytical tests for densified solid fuels

Various regions and countries have specified standard quality parameters for briquettes and pellets to ensure sustainable trade in the commodities. Typical standards for various regions are provided in Table 2.

Table 2 Densified biofuel typical properties and standard methods for property determination.

Parameter	Specification	Reference
Aspect ratio	L=~5×D	EU standards for briquettes and pellets
Moisture content	≤10%	British BioGen/UK standard
	≤18%	ÖNORM M7135/Austria
	≤8% for premium ≤10% for standard and utility	Pellet Fuels Institute standard (North American Residential/Commercial densified fuel).
Unit Density	608-768 kg/m^3	Pellet Fuels Institute standard (North America)
	≥ 525 kg/m^3	CTI-R04/5/Italy
	minimum of 1000 kg/m^3	DIN 51731/Germany and ÖNORM M7135/Austria
Calorific value	18 MJ/kg	ÖNORM M7135/Austria
	16.7 MJ/kg	British BioGen/UK
	No specification	Pellet Fuels Institute standard
Ash content	<1.5%	SS1871 20/Sweden, CTI-R 04/5, DIN 51731, and ÖNORM M7135/Austria
	≤1% for premium; ≤2% for standard ≤6% for utility grade	Pellet Fuels Institute standard (North America)
Pellet durability (Tumbling strength) Friability and Abrasion indices	≥ 96.5 for premium grade ≥ 95.0 for standard grade	Pellet Fuels Institute standard (North America)

There are few, if any, publications on standard specifications for pellets and briquettes for developing regions such as Southern Africa, perhaps due to a previously low uptake of these solid biofuels for thermochemical applications. However, the growing market and utilisation of such fuels now warrants for expeditious development of such standards. Most of the standards explored in Table 2 did not cover combustion parameters such as ignition time, burn rate and after-glow time. The burn rate and afterglow time could be functions of density, but also depend on the raw materials used. These are parameters of concern to the grilling and space heating industries where longer glowing times are desirable. While it might not be necessary to have standard testing methods for these parameters, relevant authorities may require suppliers to provide such information on the product labels. In that case, standardisation of test protocols to determine these parameters will become inevitably necessary. The same could be said for products such as charcoal briquettes with claims for cleaner burning, where smoking index or time are key parameters. In all such various use cases, specific standards or guidelines may need to be specified.

Commonly performed tests from literature, their purpose and brief protocols are reported in Table 3. These tests are reported for the manufactured briquettes only, though some of them are sometimes also performed on the raw materials.

3. Standard parameters and common analytical tests for densified solid fuels

The American Standard of Testing Materials, ASTM 2677:26T which targeted measurement of a few parameters was initially developed for charcoal briquettes. However, it was later abandoned since it did not cater for the variations that were likely to arise from different biomasses that currently dominate the briquettes feedstocks. To date, there is not yet a universally accepted series of standards adopted for biomass briquettes meant for fuel purposes, save for isolated evaluation techniques that are designed to cater for other industries such animal pelletised feedstocks. Moreover, most briquetting evaluation tests focus on the briquette thermal and mechanical properties without analysing the combustion environmental effects [12]. However, with increased advocacy on environmental pollution mitigation measures, it may be necessary to revisit the pollution assessment component in scoping the spectrum of briquette tests in future. Such tests are also important where the briquettes are for domestic use, since some researchers have often claimed that their briquettes would be less polluting, especially the charcoal ones. The use of binders potentially increases the volatile matter and ash to be produced, hence the need to empirically ascertain that the products really have lower concentrations of toxic pollutants. A few publications reported on the pollution potential by analysing the elements in the briquette from which combustion products such as oxides of chlorine and sulphur can be inferred [8]. Direct measurements of smoke index and particulate matter emitted as pollution indicators of burning briquettes were done by Pandey and Dhakal, [20] as well as Pandey and Regmi, [13]. However, briquette analysis incorporating polluting potential evaluation has been reported in limited studies. Smoke can be measured using a smoke density meter while other pollutants such as carbon monoxide and sulphur oxides can be measured using a flue gas analyser.

3.1. Physical and chemical properties

3.1.1 Proximate analysis

This is the most popular set of analytical data collected on fuels which includes the moisture content, dry matter, volatiles content, ash content and the fixed carbon content. However, because the two most common ingredients for briquetting/pelletization (coal and biomass) behave differently under heating conditions, there are variations across researchers in test conditions used. Bharti and Awasthi [56] mention the standard method ASTM D3173 for biomass proximate analysis methods applied on briquettes although in cases when biomass in the environment is regarded as waste the American Public Health Assessment (APHA) standard methods have also been applied for biomass proximate analysis. Coal analytical methods have been developed specifically from understanding coal as a fuel so most of the methods applied to densified fuels were later developed by coal related institutions. National standards such as the Indian Standard IS: 1350-1 (1984) have also been applied and cited for coal proximate analysis. The ASTM equivalents again developed for coal and coke are D-3175-02 and D3173-03 but Bharti and Awasthi [56] used this same standard to perform biomass proximate analysis. There is however a degree of uncertainty when a briquette is manufactured using a combination of coal and biomass. Adeleke et al., [8] used the coal standards when they co-briquetted coal and biomass while Ward et al. [57] used APHA methods for the proximate analysis of faecal matter-biomass co-pelletised products assessment. The use of coal based methods may be justified for carbonised biomass which has similar properties to coke [58] but when uncarbonized biomass, for example raw sawdust, is used, the analytical procedures will definitely need to be adjusted towards proven biomass based methods. In both the APHA and ASTM method cases, the machinery used may be the same but the operating parameters especially temperature, heating rates and pressure are usually different depending on whether one is dealing with biomass or coal feedstock. Some researchers chose to use their own custom operating parameters than those prescribed by the extreme two standards. Dry matter (DM) or water content analysis was conducted at 130 °C [59] with comparable results to the standard methods which specify 105 °C as temperature for dry matter analysis. Another variation to normal individual parameter tests for proximate analysis involves the use of thermogravimetric analysis TGA to determine proximate composition [60], [61] as opposed to the use of ovens.

3.1.2 Ultimate analysis

A few researchers have reported organic elemental analysis (ultimate analysis) for briquettes. Maybe this is because researchers pay more attention to the fuel properties without recognizing the polluting effects of that fuel. Pollution potential can be evaluated by understanding the volatiles content as well as the sulphur content since sulphur combusts to give the acidic sulphur oxide (SOx) gases. These SOx are greenhouse gases that also cause acid rain. The organic elemental analysis also helps in estimating the fuel heating value if the bomb calorimeter route is not available especially for domestic use of the fuel versus commercial purposes.

Table 3 Common densified fuel test parameters and procedures

Test name/parameter		Definition/Purpose	Test procedure/*Standard*	Comment on methods	Ref.
Physico-chemical properties Proximate Analysis	Moisture Content	To quantify the amount of water in the briquette. The water affects both mechanical and thermal properties.	Drying the briquettes in air/oven at 105 ˚C for 16-24hrs or until attaining constant weight then calculate the loss in weight as a ratio of initial weight. Thermo-gravimetric analysis (TGA) covers wider range of parameters *ASTM E871, ASTM D2444, ISO18134-2*	The ASTM standard is specifically for wood and wood products while the ISO standard is applicable to other solid biofuels.	[30], [31], [32], [33]
	Volatile matter	This indicates the reactivity of the biomass. High volatile matter shows high reactivity or fast burn rate but does not necessarily translate to high calorific value since at times it significantly comprises non-combustible gases.	Loss of weight on ignition at 550 ˚C or 750 ˚C depending on substrate (biomass or coal respectively). Alternatively use TGA. *ASTM D3175-18* *ISO 18123*	Volatile matter and ash content for coals and biomasses are measured at different temperatures. Confusion must be ironed out in cases involving co-briquetting.	[34]
	Ash content	To quantify the ash levels. High ash content lowers the fuel calorific value.	Procedure for volatile matter above applies to ash content also but the “LOI” temperature for volatile matter and ash analysis in coal is different. *ASTM D 1102 Standard Test Method for Ash in Wood* *ASTM D3174-12, ISO 18122*	Volatile matter and ash content for coals and biomasses are measured at different temperatures. Confusion must be ironed out in cases involving co-briquetting.	[15], [31], [35], [36]
	Fixed Carbon	To quantify the valuable carbon levels in the solid fuel. High carbon content infers higher calorific value.	The percentage of fixed carbon is obtained by subtracting the percentage sum of moisture, volatiles and ash from 100	Volatile matter and ash content for coals and biomasses are measured at different temperatures. Confusion must be ironed out in cases involving co-briquetting.	[37]
	Calorific value	To measure the heating capacity of the fuel.	Theoretically determined through established empirical correlations such as Dulong, Demirbas, etc. OR use bomb calorimeter procedures. *ASTM D5865-13, ISO 18125*	The ASTM method focuses on coal and coke while the ISO standard emphasizes biofuels. There is need for harmonization.	[30], [37], [38], [39]
Ultimate Analysis	CHNS	Measuring the organic elements in the briquette. These help in deducing calorific values.	Elemental analysis procedures used, and oxygen is determined by difference. *ASTM D3176-15 (C, H, N, S)* *ISO 16948 (C, H, N)* *ISO 16994 (S)*	The ASTM method focuses on coal and coke while the ISO standard emphasizes biofuels. There is need for harmonisation.	[40], [41], [42], [43]
Density	Compressed density and Compaction ratio	High compaction ratio signifies low void spaces in product hence better storability and transportation characteristics.	Compressed density is measured just after producing the densified product. Compaction ratio is calculated as ratio of compressed density to substrate powder initial density. *ASTM D2395, ISO 18847*	The ISO standard generalises its application to densified materials though the ASTM emphasizes on wood.	[25], [44], [45]
	Relaxed density and Relaxation ratio	High relaxed density and low relaxation ratio implies a more stable briquette.	The ratio of compressed density to relaxed density give the relaxation ratio. Relaxed density is measured after 30 days from manufacturing date. *ASTM D2395, ISO 18847*	The ISO standard generalise its application to densified materials though the ASTM emphasises on wood.	[25], [44], [45]

Table 3 (cont.) Common densified fuel test parameters and procedures

Test name/parameter		Definition/Purpose	Test procedure/*Standard*	Comment on methods	Ref.
Mechanical properties	Tensile/ Compressive strength	Measures the maximum load a briquette takes before it cracks giving an indication of resistance to crushing forces. For comparability the resistance is determined as the ratio between the breaking force and cross-sectional area (N/mm^2).	Universal Compressive or Tensile testing machine in axial and radial directions respectively. *ASTM D2166-85*	The ASTM method emphasises on wood. Applicability to other potential feedstocks is not addressed. There are other manual methods such as triangular model of determining maximum height of stack	[46], [47]
Assessing resistance to fragmentation	Impact resistance index (IRI) Shatter index (SI)	Give indications of briquette's resistance to impact forces.	Drop shatter tests measures number of pieces (or particle size distribution) formed on dropping briquettes from a predetermined height for a recorded number of times. *ASTM D440-86; ASTM D440-06 for coal; ISO 616*	Specific to coal without catering for other feedstocks. This leaves uncertainty on adequacy of protocols for biomass-based briquettes.	[2], [12], [48], [49], [50]
	Tumbling strength index Friability index	Measures the briquette resistance to abrasive forces.	In the tumbler test, briquettes are loaded into a tumbling drum then particle size distribution and/or weights assessed after tumbling under predetermined conditions	Several variants of this test exist and standardization for briquettes is required	[51], [22]
	Water resistance index (WRI) Porosity index (PI)	Assesses the briquette's resistance to absorb water when exposed to this media during storage or transportation.	Briquettes soaked in water for 30 minutes then initial and final weights of the briquette are used to calculate % water absorbed. This is subtracted from 100 to get WRI. *ASTM D870-15*	The ASTM standard method cited here was developed for coatings and may need modifications to suit fuel briquette purposes.	[2], [12], [52].
Combustion characteristics	Burning rate	Evaluates how fast a fuel burns	A known amount of fuel is burnt at standard temperature and pressure then the time taken to use up that fuel is recorded.	Several tests as stated on the Clean Cooking Alliance website. https://cleancooking.org/	[29]
	After glow test	The time taken during briquette glow after the fire goes off	A stopwatch is used to record the time when the glow will still be observable by a naked eye.	No international standard method exists	[19], [53]
	Ignition time	The time it takes a for briquette to catch a fire	The briquettes are ignited by use of a gas burner or liquid (kerosene, ethanol) fire until they catch the fire.	No international standard method exists	[54]
	Water boiling test	The time taken to boil water	The time taken to boil a given amount of water	Several versions exist as described on Clean Cooking Alliance website.	[55]
	Heat utilization efficiency	Measures the fuel cooking efficiency. Also called thermal efficiency. The specific fuel consumption can also be calculated.	A burning fuel is used to heat water until it starts to boil. The amount of fuel used to attain the water boiling temperature is recorded per amount of water boiled or per unit time. *ISO 19867-1: 2018*	The standard is fairly new and therefore may be revised extensively in future if there are shortcomings identified in these early years of adoption.	[13], [29], [54]

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Some researchers have resorted to empirical expressions based on proximate and ultimate elemental analysis to determine the fuel heating values, in the absence of a bomb calorimeter [30], [62]. These correlations, which are convenient and cheaper to use, but not as accurate as the bomb calorimeter, have been extensively reviewed elsewhere [30], [62], [63]. The standard EN15104:2011 has been used on biomass [64] to acquire the CHNS profile though the ASTM D5373:2016 serves the same function and is specifically designed for coal. It is then confusing which of the two standards should be followed in the case of a biomass-coal mixture briquette.

3.1.3 Density and related ratios

The calculations for relaxed density therefore relaxation ratio are performed after different curing times by different researchers, and this makes it difficult to compare briquettes on the market. For instance, Aransiola et al. [25] used thirty days curing while Muazu et al. [53] allowed a curing time of one day only before performing analysis. Also, there is need to standardize definitions; for instance, some authors such as Onukak et al. [65] have referred to bulk density as the density of one briquette, probably since it is an agglomeration of many individual biomass particles. Others refer to this density as relative density, while others termed it relaxed density. The problem comes when trying to define the density of a group of briquettes, since bulk density would still be a relevant terminology. The use of 'relative' or 'relaxed' density for a single briquette therefore seems less confusing.

3.2. Mechanical properties

3.2.1 Tensile/compressive strength

From the reviewed articles, most researchers chose to measure and report either tensile or compressive strength but rarely both parameters. The difference between these two pertains to the orientation of the briquette during the testing especially for cylindrically shaped briquettes. Compressive strength applies when the force is applied to the briquette tangentially the force is applied radially for tensile strength test. The two orientations yield different results and [12] recommends testing briquettes in their weakest orientation which is the tensile strength [66]. It is further emphasized that the tensile strength must be expressed as stress rather than force units so that the shape and size of briquettes does not affect comparison of briquettes based on this parameter [12]. Despite this recommendation a recent research study reported compressive strength in force (N) rather than stress (N/m^2 or N/mm^2) units [67]. What also tends to vary among researchers are the machines used and operating settings such as the rate of force application on the briquette, with researchers such as Dohm et al. [27] recommending an improved manual test stand for precise results comparable to automated test stands recommended by Dragusanu et al. [9]. This is a knowledge gap that needs to be closed. There is limited reference in literature on the threshold points below which the strength should be said to be unqualified, warranting the rejection of briquettes. The feeling may be that, since briquettes made from artisanal hand operated machines are often for domestic applications at small scale operations, they may not need to go through all these rigorous yet subjective tests. However, considering the increasing global demand for biomass briquettes and pellets also in the interests of consumer satisfaction, it is still critical to have standard tests and parameters [68], [25].

3.2.2 Other assessments on briquette mechanical integrity

There seem to be a general consensus among researchers on using the drop tests for assessing impact resistance, use of the tumbling test for evaluating resistance to abrasive forces and water absorption test to ascertain durability in moist environments. However, there are inconsistences picked in literature regarding the application of each of these techniques. A few examples of works picked and reported in Table 4 show some of these inconsistences.

A few researchers such as Rath et al [69] also conducted Rockwell hardness tests, a parameter meant to measure a material's resistance to permanent deformation when subjected to scratching, bending or cutting. Dragusanu et al. [9] mention a customised test to determine the splitting strength of briquettes using a cutting knife of a specified rounding radius, thickness, width and tip angle, driven by a universal compressive strength tester.

3.3. Thermal and Combustion properties

The calorific value or high heating value (HHV) of fuels is a physical property affected by temperature, frequently presented long with proximate compositions. The standard test for calorific value uses the bomb calorimeter (ASTM D5865-04) although estimations from proximate and ultimate analysis may also suffice in the absence of bomb calorimetry equipment. The procedures for bomb calorimetry are standardised for all fuels regardless of densified or raw fuels and no inconsistences have been observed in applying this technique besides the choice of performing the test or not performing it [37].

In most cases combustion properties other than the calorific value are not reported for densified fuel analysis in literature. This is despite that the analytical equipment and techniques are easily conducted at minimal costs. One major reason for not performing combustion tests among researchers could be that most of the tests involve appreciable human judgment or errors, therefore, researchers find these tests to be subjective for application as standard procedures. However, the information derived from such tests would be very useful especially if controls are put in place to reduce the human error issues.

The risk of such errors can be reduced by performing inter-laboratory tests on the same samples and using one experienced person in testing the fuels that should be compared in each independent laboratory. Intra-laboratory differences reported by one analyst from one laboratory should be given attention only if there are matching inter-laboratory conditions in the case of combustion property evaluations. Minor inter-laboratory variations should not warrant further investigations. A few inconsistences picked among practitioners on briquettes combustion properties evaluation are discussed below.

3.3.1 Burn rate, Open air combustion test and Water boiling test

These combustion-related tests were performed separately by some researchers while others performed them in one set up. The open air combustion test is performed by burning fuel in the open air [53]. Kizito et al. [15] combined the burn rate and water boiling tests and in this set up, it would be a misnomer to call this set up an open air combustion test although data accrued from this study can still be used to compute the burn rate just as in the case of an open air combustion set up. In these analytical procedures, the amounts of biofuel used in the experiment as well as the initial water quantity to be boiled and presentation of results varied across researchers. Bonsu et al. [76] conducted the tests starting with about 1kg fuel and 100ml of water while Ajiboye et al. [3] used a starting fuel quantity of 0.1 kg for the same amount of water. Another inconsistency among researchers in performing combustion tests involves the weight and temperature data recording and timing intervals. While most researchers collected final time duration and weights after the fire burnt out [77], others collected data at time intervals throughout the burning period [13], [76]. Specifically for the water boiling test, the amount of water used and type of stove and kettle/pan also tended to be different across researchers.

3.3.2 Ignition time

A major inconsistency in the application of this test is around the use of different fuels such as ethanol [15], propane gas powered torch [53], kerosene [16] to ignite the briquettes. The different fuels introduce variations in fire spread and they are also different in calorific values. These variations have potential to distort ignition time comparisons across differently formulated briquettes.

Table 4 Common densified fuel tests and procedures

Test/(Variable)	Major inconsistences across different studies	References
Drop test	Authors report different starting heights and the number of times the briquettes must be dropped as well as the quantity of briquettes tested. Some mention that the surface roughness on which the briquette is falling also matters while others ignore the floor conditions.	[12], [61], [70], [71]
Impact resistance index (IRI)	Some studies report the strength index (SI) not the IRI and these two parameters are computed differently.	[2], [12]
	The IRI is calculated using different formulae in different publications. One formula divides the number of drops by the height while other formulae do not divide by the height.	[12], [71]
Tumbling test	Use of the friability index instead of the common tumbling strength index used by other researchers. The two indices are calculated differently. The friability index uses one piece (final mass divide by initial mass) while the tumbling uses a cut off size class mass measurements arising from several particles and several briquettes.	[22], [51]
	The operating conditions vary across researchers. For example, amount of sample tumbled, duration of tumbling, rotational speeds and sieve mesh sizes used as cut off points.	[72], [73]
	The configuration/orientation of the rotating drum varies from one experiment to the other with vertical and horizontal configurations having been employed in different cases. Instead of a drum a tumbling box can also be used.	[12], [74], [75]
Water absorption test	In most cases, briquettes weight was measured after immersing them in water. The WRI was then calculated from weights before and after immersion. The duration of soaking varies across researchers ranging from 30s to 2 mins.	[5]
	In a different calculation, only the absorption rate is calculated by dividing the weight differences above with the duration of immersion. No WRI is calculated in this case. In some cases, the PI is calculated instead of the WRI	[2], [75]
Water resistance index (WRI)	Water resistance measured as the time taken for onset of briquette dispersion when immersed in water at room temperature. No weights measurement as in the WRI procedure.	[5].
Porosity index (PI)	Water resistance involved immersing briquette in water for 8 hours before curing and testing its compressive strength thereafter. This is opposed to the WRI route. Another researcher followed the same procedure but soaked for 2 hours instead.	[70], [72]

3.3.3 After glow

Though the definition of afterglow is consistent among researchers, there are variances in how the test procedure is conducted, for example, Muazu and Stegemann [53] determined the time after the flame disappeared on its own but Moki et al., [19] had to blow off the flame while it was still burning instead of waiting for it to go off by itself.

3.3.4 Specific fuel combustion or Thermal efficiency

Different amounts of water have been reportedly used in water boiling test experiments. Some researchers prefer to report this as thermal efficiency (Equation 1) instead of the specific fuel consumption (Equation 2). The thermal efficiency calculation however requires predetermination of more parameters [13] such as the temperatures of the ambient, the fire and the vessel in which the water is boiling. The calculations also require the amount of water evaporated to be determined including knowing the specific heat capacities of the water, the latent heat of evaporation of the water and the calorific value of the fuel.

$$\theta = Mw\ x\ Cw\ x\ \Delta T + Me\ x \frac{L}{Mf\ x\ Hf} \qquad Equation\ 1$$

Where θ thermal efficiency
M_w initial weight of water (kg)
C_w specific heat capacity of water KJ/kg·°C)
ΔT temperature rise in the water (°C)
Me mass of water evaporated (kg)
L water latent heat of evaporation (KJ/kg)
M_f Amount of fuel burned (kg)
H_f Fuel calorific value (KJ/kg)

$$S = \frac{F}{W} \qquad Equation\ 2$$

Where S Specific fuel consumption rate
F Amount of fuel burned to attain boiling (kg)
W Amount of boiling water (kg)

3.4. Infrequently reported densified fuel parameters and analytical techniques

Surface roughness assessed visually [11], [16] or by use of scanning electron microscopy (SEM) [10], [78], [79] and surface weathering assessed using QUV Accelerated Weathering Tester in conjunction with attenuated total reflection spectroscopy (ATR) [61] are seldom tested densified fuel parameters. The surface assessment helps to infer ease of combustion as the heat and oxidant enter the briquette [79]. The parameter must possibly correlate with briquette density. Brunauer-Emmett-Teller (BET) surface area, in particular, is more frequently reported in research articles where the application of carbonized briquettes is adsorption [80]. Surface roughness may also give information regarding challenges in transportation on conveyor belts and is therefore only important in large operations that involve these conveying systems as opposed to artisanal scale operations that instead use bags for transportation of briquettes. The weathering test is performed in view of testing possible binder degradation effects over time if the binders are meant to be stored for longer periods in the open atmosphere. The reactivity index (RI), which indicates how well a briquette will react with other oxidizing agents in a combustion process [16], [51], is another briquette parameter that has not received substantial attention from researchers possibly because of the insignificance of the information it conveys. This parameter may only be critical in large commercial applications such as blast furnaces or boilers. Briquettes destined for household use, which seem to be the main application of briquettes currently, are not likely to have interference from other oxidizing agents and consequently, they are exempted from this analysis in most cases.

Kaur et al. [11] analyzed for a parameter that they called density hardness as well as colour. The procedures around this were not clear and the significance of such tests/parameters have not been justified elsewhere. These parameters are rarely reported by many researchers who have performed briquette evaluations. It is therefore necessary to investigate these anomalies and address them when standardizing briquette evaluation methods and techniques.

Borowski et al. [36] reported the burn-up factor which was described as the share of burned fuel relative to total dry matter. These researchers also determined time of smoke, firing up time, burning time of temperature above 180 ˚C as well as maximum temperature. Firepower which is defined as the average power output of the stove during the water boiling test was also assessed by Sawadongo et al. [37]. This parameter is used to characterise briquettes combustion properties, though it is rarely used with the traditional solid fuels such as wood and coal. This parameter is used to characterise briquettes combustion properties, though it is rarely used with the traditional solid fuels such as wood and coal. Burn-up factor and firepower are not normally reported by most briquette researchers despite conveying important information regarding fuel performance.

Thermogravimetric analysis (TGA), though not used by many researchers, gives both the thermal stability and proximate analysis data in one experiment [61]. Most probably the researchers who do not use this technique lack the necessary facilities. Using the TGA or the traditional oven-muffle furnace routes should give ideally the same accuracy in data collected but with minimal steps in the TGA option.

4. Overview and future outlook of analytical methods for densified fuels

The densified solid fuel industry has been expanding rapidly to meet both environmental goals and growing energy demand. As in other cases of rapid growth, standardization and regulation of protocols have lagged behind and this written account identifies the grey areas. The techniques and protocols used to date in such grey areas have helped researchers compare the efficacy of various binders, raw materials, machines and procedures within the same set of parameters. A few such tests were replicated and protracted to other variables by other researchers with some concerns over the constancy of other environmental parameters. The future, however, demands uniformity, statistically robust replicability, dependability and standardisation of test protocols for determining the quality of densified solid fuels, as they become a widely accepted and used sustainable commodity. Accredited business management systems stipulate standardization of procedures as a good practice which triggers accrual of many benefits for commercial scale business operations. It is therefore recommended that players in the solid fuels densification industry focus on standardizing briquette testing protocols. It is important to come up with categories of briquettes and their associated quality parameters based on applications and these may then vary slightly from country to country or region to region, depending on the specific infrastructure largely used. Spelling out quality parameters is the easier part; there is need to agree on standard protocols for testing these parameters especially for impact resistance, burn rate, burn time and water resistance. Test protocols for proximate, elemental and calorific value analyses are largely standardised. For some procedures such as compressive strength, there may be need for an increased rate of technology transfer so that requisite standard equipment can be used in developing regions that are fast becoming an important source of densified fuels. Standardization, lessons can be borrowed from the biogas industry that has recently taken the same initiative to standardize its biomethane potential testing protocol for that industry [81], [82], [83]. However, for the artisanal briquette producers who normally reside in low-income rural communities, some of the rudimentary non-standard procedures reviewed in this paper will remain in use for the foreseeable future, especially for local developing regions that do not have any standardised parameters for these products. However, such circumstances short-change customers especially where there are exaggerated claims on calorific values and smoking indexes. Not to mention losses incurred during transportation and storage for underspecified products. The imminent expansion of this industry calls for a quick action in developing necessary standards and protocols even for such developing regions, for both local use and the target export market. The industry also has to take advantage of technological advances in analytical equipment to fast track and improve accuracy of analytical data. Use of TGA versus the muffle furnace routes for proximate analysis is recommended since the risks of human errors are higher for the latter case. However, it is also argued that for volatile matter analysis of biomass, the TGA method requires some minor modifications when shifting from coal to biomass. This is not the case with the oven methods which are applicable without modification to both coal and biomass [84]. Another example is the choice of elemental analysis route where EDX coupled with SEM images for more information, or sticking to the traditional ASTM elemental analyser route, are available options. The EDX data will also disclose the potentially polluting elements such as chlorine and sulphur. Inclusion of the often-neglected parameters which are however easy to perform and may transmit information related to environmental sustainability such as time of smoking, firing up time, etc. It would be difficult though to agree on the cut off levels for these parameters which are highly dependent on the raw material used. However, for purposes of building a database of knowledge for future referencing, the analytical steps must be standardized and results reported in publicly accessible research articles. As this database and practice builds up, feedback articles from commercial trials can then also contribute to revisions and ruggedization of these standards.

5. Conclusions

A detailed critical review of analytical methods used for densified biofuels has been presented. It has been noted that there are inconsistencies in the application of tests and techniques with some researchers choosing to omit certain tests in their studies or including tests that duplicate the information conveyed by other parameters. To arrest these anomalies especially in commercial briquette making practice, authors recommend standardization of some of the important tests such as calorific value determination, mechanical strength, water boiling tests as well as those tests that convey information on pollution potential of the briquette when combusted. Such critical parameters would then need specifications of minimum thresholds for specific application areas. Other ‘non-critical’, yet competitive parameters as ignition time, burn rate, after-glow time and smoking index/time that may be of concern to the grilling, space heating and clean-energy industries may not necessarily have threshold specifications or standards. However, relevant authorities may require suppliers to provide such information on the product labels to ensure that users are informed of the product specifications. The methods used to determine these parameters still need to be standardised to allow for uniform comparisons and protect consumers from being short-changed. In standardising, considerations for inclusivity can be made by adopting cheap, easy-to-perform protocols methods for assessing the densified solid fuels such as those used in the widespread artisanal fuel densification practices. This, along with knowledge and technology transfers, will allow for the widespread participation of developing regions that could be an important supplier of densified fuels in the near future.

Author contributions

Conceptualisation and manuscript writing were performed by Gratitude Charis and Charles Rashama. Gratitude Charis and Charles Rashama wrote the first draft of the manuscript. Reviews and corrections of the first draft were carried out by Bilal Patel and Benias Nyamunda. All authors read and approved the final manuscript.

Declarations

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