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Preparation procedures of food and beverage samples for oxygen bomb calorimetry: A scoping review and reporting checklist

Zane Hopper [a,b,c,*], Ben Desbrow [a,b], Shelley Roberts [a,b,c], Chris Irwin [a,b]

[a] School of Health Sciences and Social Work, Griffith University, Gold Coast, QLD, Australia
[b] Menzies Health Institute Queensland, Griffith University, Gold Coast, QLD, Australia
[c] Gold Coast Hospital and Health Service, Gold Coast, QLD, Australia

Abstract

Standardised bomb calorimetry methods are essential to accurately quantify the gross energy within food and beverages, yet no accepted protocols exist. The objective of this review was to synthesise literature on food and beverage sample preparation methods used for conducting bomb calorimetry. This synthesis enhances our understanding of the extent to which methodological variances may currently affect estimates of the caloric values of dietary items. Five electronic databases were searched for peer reviewed literature on food and beverage energy measurement via bomb calorimetry. Data were extracted on seven identified methodological themes, including: (1) initial homogenisation, (2) sample dehydration, (3) post-dehydration homogenisation, (4) sample presentation, (5) sample weight, (6) sample frequency, and (7) equipment calibration. A tabular and narrative approach was used to synthesise the data. Studies that specifically explored the impact of any methodological variance on the energy derived from foods and/or beverages were also considered. In total, 71 documents describing food and beverage sample preparation techniques and processes used for bomb calorimetry were identified. Only 8% of studies described all seven identified sample preparation and calibration processes. The most frequent approaches used included: initial homogenisation − mixing or blending (n = 21); sample dehydration − freeze drying (n = 37); post-dehydration homogenisation − grinding (n = 24); sample presentation − pelletisation (n = 29); sample weight − 1g (n = 14); sample frequency − duplicate (n = 17); and equipment calibration − benzoic acid (n = 30). The majority of studies that have measured food and beverage energy via bomb calorimetry do not describe sample preparation and calibration methods in detail. The extent to which different sample preparation processes influence the energy derived from food and beverage items is yet to be fully elucidated. Use of a bomb calorimetry reporting checklist (described within) may assist with improving the methodological quality of bomb calorimetry studies.

Keywords: Bomb calorimetry, Food, Gross energy, Sample preparation

1. Introduction

Bomb calorimetry is used to quantify gross energy (i.e., total chemical energy) released from the complete combustion of products. The process involves igniting a sample (liquid or solid) under stable temperature conditions and measuring calorific values from the resultant change in temperature [1]. The technique is commonly used to evaluate energy efficiency and product quality of fossil fuels and biomasses [2]. In nutrition science, the process can be employed to quantify the gross energy content of food and beverages [3], offering a method to verify energy values within dietary analysis databases as well as those displayed on food nutrition information labels.

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* Corresponding author at: School of Health Sciences and Social Work, Nutrition and Dietetics, Griffith University, Building G40 Level 2, Gold Coast, QLD, Australia.
E-mail addresses: zane.hopper@griffithuni.edu.au (Z. Hopper), b.desbrow@griffith.edu.au (B. Desbrow), s.roberts@griffith.edu.au (S. Roberts), c.irwin@griffith.edu.au (C. Irwin).

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To ensure bomb calorimetry is performed accurately, standardised methodological procedures are required. Several published international standards exist for undertaking bomb calorimetry to determine gross energy (i.e., ISO 1928, ASTM D5865, AS 1038.5, BS 1016, DIN 51900) [4–8]. However, these methods have been primarily established for natural resources such as coal and crude oil. Preparation methods for these fuels may not include critical steps needed for analysis of food and beverages. For example, many foods require homogenisation and dehydration to ensure the small sample used is completely combustible and representative of the entire product. To date, researchers have employed a variety of sample preparation techniques and equipment calibration processes when undertaking bomb calorimetry for food and beverage energy measurement. These include freeze drying [9], oven drying (at various temperatures) [10,11], grinding [12], mixing [13], combustion of different sample volumes [14,15], and analyses of varying sample sizes [16,17]. The extent to which these different approaches have been used, and their impact on subsequent energy discernment, is yet to be rigorously examined.

The aim of this investigation was to describe the variety of methodological approaches used to prepare food and beverage samples for gross energy determination via oxygen bomb calorimetry. This information will provide insight into the heterogeneity of sample preparation methods and facilitate the development of more consistent sample preparation and equipment calibration procedures for the measurement of food/beverage energy via bomb calorimetry.

2. Methods

2.1. Protocol and registration

This study was conducted in accordance with the Preferred Reporting Items for Systematic Reviews and Meta-Analyses extension for Scoping Reviews (PRISMA-ScR) guidelines [18]. The scoping review protocol was registered in the Open Science Framework (OSF) register of scoping reviews (Registration DOI https://doi.org/10.17605/OSF.IO/JFHDT).

2.2. Eligibility criteria

Eligibility criteria for studies were based on the PICOS (population, intervention, comparison, outcome, setting/design) method (Table 1). Investigations must have used bomb calorimetry to measure the gross energy content of food and/or beverages habitually consumed by humans. This included pre-prepared foods (e.g., items from supermarkets, restaurants and/or takeaway providers), as well as animal-based foods (e.g., fish, chicken, lamb, pig). Some animal-based studies included the whole carcass (i.e., head and bones) in the analysis, which may not typically be consumed by humans. However, such studies were included to achieve a greater representation of food samples for the review. There was no requirement for a control group. Only original research presented as full-text papers written in English were included, without restriction on study design or location. Papers that did not provide any detail of sample preparation, analysis or equipment calibration processes were excluded.

2.3. Search strategy

For this scoping review, five major electronic databases (Scopus, Web of Science, PubMed, Royal Society of Chemistry, AGRICOLA of USDA) were searched in May 2022 using a systematic search process with the main field search terms ‘bomb calorimetry’ and ‘energy’ (see Supporting Information, File S1 (https://www.jfda-online.com/journal/vol31/iss2/3/)). All identified citations were collated and duplicates removed. Title/abstracts were screened using the eligibility criteria. Reference lists of included papers were also screened to identify other eligible papers that were not captured in the original search. Full-text screening of eligible publications was completed independently by one author and any uncertainty was resolved in consultation with the research team.

Table 1. Eligibility criteria of peer reviewed literature for inclusion in the review.

Inclusion criteria	
Population	Food and beverages habitually consumed by humans
Intervention	Bomb calorimetry testing, AND INCLUDING details on reported sample preparation techniques AND/OR number of samples analysed AND/OR equipment calibration processes
Outcome	Gross energy measurement
Study design	Primary research using any observational or experimental study design OR studies using quantitative, qualitative, or mixed methods data collection

2.4. Data extraction and synthesis

Extracted data included: document identifiers (author, year, DOI), bomb calorimetry sample type (food type), bomb calorimeter name/brand, characteristics of sample preparation methods (freeze drying, dehydration, weight), and equipment calibration processes. Methods were grouped according to seven identified sample preparation and equipment calibration themes identified from bomb calorimetry manufacturer guides/manuals [1,19–21] and international calorimetry standards [4–8]: (1) initial homogenisation, (2) sample dehydration, (3) post-dehydration homogenisation, (4) sample presentation, (5) sample weight (mass), (6) sample frequency, and (7) equipment calibration. When studies were specifically conducted to compare different methods (e.g., freeze drying vs oven drying), data were only extracted for the reference method. A tabular and narrative approach was used to synthesise data. Findings were organised in a sequential fashion determined by characteristics of individual sample preparation methods and equipment calibration processes employed.

3. Results

3.1. Study selection

Following the electronic database search and subsequent synthesis of articles (Fig. 1), 71 studies were included in the final review [3,9–17,22–52, 53–82]. Full details of these investigations are provided in the Supporting Information_2 (https://www.jfda-online.com/journal/vol31/iss2/3/). Thirty-four studies were excluded due to failing to provide effective methodological detail on the energy measurement or calibration process. One study was excluded on the basis that it was not published in English (Portuguese).

3.2. Bomb calorimetry sample preparation techniques

A summary of bomb calorimetry sample preparation techniques employed in the 71 studies is outlined

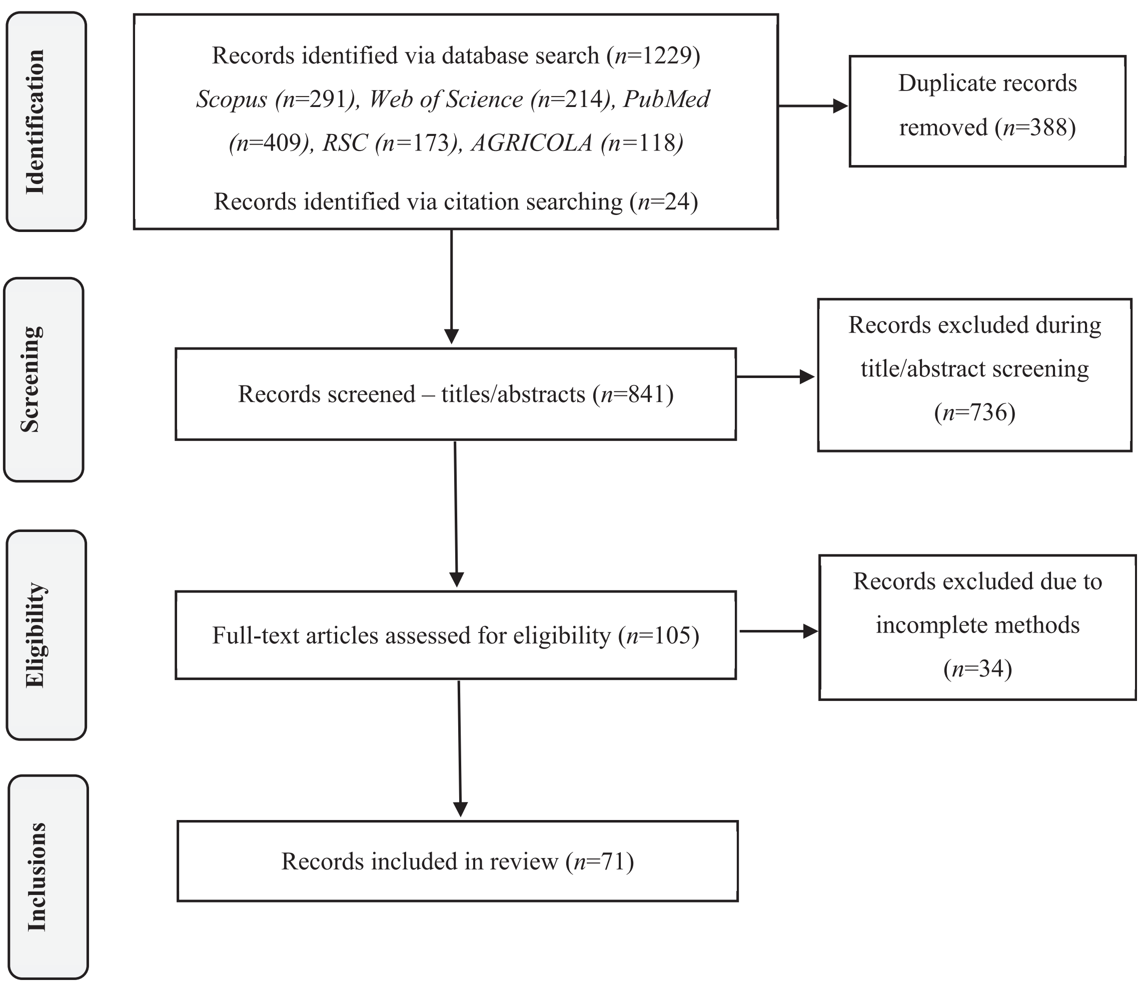

Fig. 1. Flow diagram of included studies.

Table 2. Sample preparation techniques used in bomb calorimetry studies of food and beverages.

Phase	Preparation method ($n = 71$)	Sample structure	# Studies reporting method	# Studies reporting use of equipment/process
1	Initial homogenisation	Liquid ($n = 7$)	Homogenised ($n = 1$)	Blended ($n = 1$)[a]
		Semi-solid ($n = 30$)	Homogenised ($n = 26$)	Blended ($n = 12$)[b,c]
				Mixed ($n = 1$)[d]
		Solid ($n = 34$)	Homogenised ($n = 17$)	Blended ($n = 7$)[e,f]
				Cut up ($n = 4$)[g]
				Cut up & ground ($n = 2$)[h]
			Nil ($n = 10$)[j]	Minced ($n = 1$)[i]
2	Sample dehydration	Liquid/Semi-liquid	Freeze dry ($n = 37$)	−50 to −190°C ($n = 6$)[k]
				3 days ($n = 2$)
				7 days ($n = 1$)
			Oven dry ($n = 25$)	15 to 85°C ($n = 23$)[l]
				For up to 72 h ($n = 14$)[m]
				Constant weight ($n = 7$)
			NA ($n = 1$)[n]	
3	Post-dehydration homogenisation	Solid (all)	Homogenised ($n = 33$)	Ground ($n = 24$)[o]
				Mixed/blended ($n = 6$)[p]
				Macerated ($n = 1$)[q]
			NA ($n = 2$)[r]	
4	Sample presentation	Varies	Pellet ($n = 29$)	
			Saw dust ($n = 1$)	
			Paste − Benzoic acid ($n = 1$)	

[a] Commercial food blender.
[b] Commercial food blender ($n = 4$); food processor ($n = 1$); not stated how ($n = 7$).
[c] Mixed with water ($n = 6$).
[d] Not stated.
[e] Commercial food blender ($n = 1$); not stated ($n = 6$).
[f] Mixed with water ($n = 3$).
[g] Cut into 0.5 cm cubes ($n = 1$); 2.5 cm cubes ($n = 1$); 'thin' slices ($n = 1$).
[h] Milling machine ($n = 1$); grinding machine ($n = 1$).
[i] 5 mm sieve machine ($n = 1$).
[j] Nil homogenisation due to sample being dried first.
[k] −50 °C ($n = 1$); −60 °C ($n = 1$); −77 °C ($n = 2$); −84 °C ($n = 1$); −190 °C ($n = 1$).
[l] 15 °C ($n = 1$); 50 °C ($n = 1$); 55 °C ($n = 3$); 60 °C ($n = 9$); 70 °C ($n = 6$); 80 °C ($n = 1$); 85 °C ($n = 2$).
[m] 1 h ($n = 1$); 8 h ($n = 1$); 12 h ($n = 1$); 20 h ($n = 1$); 24 h ($n = 1$); 48 h ($n = 4$); 72 h ($n = 5$).
[n] Nil liquid removal due to sample analysed (olive oil) using carrying agent (saw dust).
[o] Coffee grinder ($n = 4$); mortar and pestle ($n = 4$); milling machine ($n = 2$); crushed ($n = 1$); not stated ($n = 13$).
[p] Electric mixer ($n = 2$); food processor ($n = 2$); not stated ($n = 2$).
[q] Not stated.
[r] Nil homogenisation due to sample (olive oil, alcohol) using carrying agent (saw dust) ($n = 1$); sample comprising dry pasta ($n = 1$).

in Table 2. Across all studies, seven (10%) analysed liquids,[1] 30 (42%) analysed semi-solids,[2] and 34 (48%) were conducted on solid[3] samples [83]. Eight different bomb calorimetry machines were reported in the analysis. The majority of these (44; 62%) stated manufactured by the Parr Instrument Company and comprised both 'wet' and 'dry' systems.

3.2.1. Phase 1 − initial homogenisation process

Of the 71 studies, 44 (62%) reported undertaking an initial sample homogenisation process, while ten (14%) did not undertake homogenisation (whole dried fish (n = 9) and nuts (n = 1)). The remaining 17 (24%) studies did not specify if a homogenisation process was completed. Of the studies that did use an initial homogenisation process, 21 (48%) describe blending or mixing samples. When blending or mixing was employed, nine reported adding water (volume used remained undescribed). Seven studies indicated the initial homogenisation involved samples being 'cut up' or minced. The remaining 16 (36%) studies that reported an initial homogenisation process, failed to provide any detail of how this was conducted.

[1] Liquid − flows freely and is not a solid, e.g., water or oil.
[2] Semi-solid − highly viscous; slightly thick, e.g., soft fruits and vegetables, mixed diet.
[3] Solid − not a liquid or gas; hard or firm, e.g., beef, chicken, fish, nuts.

Table 3. Sample weight used in bomb calorimetry studies of food and beverages.

Sample weight (g)	Number of studies ($n = 71$)	Sample type
1–2	1	Soft drink ($n = 1$)
1	14	Mixed diet ($n = 8$)
		Animal ($n = 4$)[a]
		Bakery ($n = 1$)
		Food crops ($n = 1$)
0.5–1	3	Infant formula ($n = 2$)
		Food crops ($n = 1$)
0.02–0.4	7	Animal ($n = 7$)[b]
Not stated	46	Mixed diet ($n = 17$)
		Animal ($n = 11$)[c]
		Mixed – Restaurant ($n = 5$)
		Human milk ($n = 4$)
		Nuts ($n = 5$)
		Bakery ($n = 1$)
		Banana ($n = 1$)
		Olive oil ($n = 1$)
		Pasta ($n = 1$)

[a] Sample type analysed: Fish ($n = 3$), Lamb ($n = 1$).
[b] Sample type analysed: Fish ($n = 7$).
[c] Sample type analysed: Fish ($n = 4$), Shellfish ($n = 4$), Pig ($n = 1$), Chicken ($n = 1$), Goat ($n = 1$).

3.2.2. Phase 2 – sample dehydration

Sixty-two (87%) studies reported using a liquid removal process, with 37 (60%) freeze-drying and 25 (40%) oven-drying. Details of the temperature and duration of the drying procedures are described in Table 2. One study did not require liquid removal (i.e., involved olive oil placed directly onto sawdust substrate [45]). The remainder of studies (n = 8) did not specify if this process was undertaken.

3.2.3. Phase 3 – post-dehydration homogenisation

The majority of studies (38; 54%) did not report undertaking a post dehydration homogenisation process. Of the 33 (46%) that did, 24 (73%) reported grinding the dehydrated sample, while the remainder were either mixed or macerated. Two studies did not require this process (olive oil placed directly onto sawdust substrate [45] and dry pasta [77]).

3.2.4. Phase 4 – sample presentation

Most studies (40; 56%) failed to report if a specific sample presentation approach was employed prior to combustion. The remaining 31 studies primarily employed pelletisation (29; 94%), while one study combined the food sample with sawdust [45] and another combined the sample with a benzoic acid paste [71].

3.2.5. Sample weight

A summary of bomb calorimetry sample combustion weights is outlined in Table 3. Of the 71 studies, 46 (65%) did not report a combustion sample weight. Of those that did, 14 (56%) used a 1g sample. All seven studies using very low sample weights (≤0.4g) analysed the energy content of fish.

Table 4. Sample analysis frequencies used in bomb calorimetry studies of food and beverages.

Sample analysis frequency	Number of studies ($n = 71$)
Single	1
Duplicate	17
Duplicate/triplicate	13[a]
Triplicate	7
Quadruplicate or more	4[b]
Not stated	29

[a] Triplicate if variance was 0.5 to <2% ($n = 2$), 2–5% ($n = 7$) or >0.03 kcal ($n = 4$).
[b] Quadruplicate ($n = 2$), Five ($n = 1$), Twenty ($n = 1$).

3.2.6. Sample frequency

A summary of sample analysis frequencies is outlined in Table 4. Of the 71 studies, 42 (59%) reported a sample analysis frequency. Of these, 17 (40%) reported analysing in duplicate, while 13 (31%) reported using triplicate analysis if the variance in duplicate samples was between 0.5 and 5%. Seven (17%) studies used triplicate analysis and five (12%) conducted quadruplicate or greater analyses on samples.

3.2.7. Equipment calibration – method and frequency

A summary of equipment calibration methods and frequencies is outlined in Table 5. Of the 71 studies, 39 (55%) did not list a calibration method. Of those indicating a method, the majority (30; 94%) used benzoic acid as the calibration standard. A calibration frequency was only listed in eight (11%) studies, with the most common of these (50%) reported as occurring after every ten combustions.

3.3. Studies with incomplete methodology reporting

A summary of the studies reporting bomb calorimetry methods is outlined in the Supporting

Table 5. Machine calibration methods used in bomb calorimetry studies of food and beverages.

Calibration method	Number of studies ($n = 71$)	Frequency
Benzoic acid	30	Every 10 samples ($n = 4$)
		Every 20 samples ($n = 1$)
		Daily ($n = 3$)
		'Other' not defined ($n = 2$)
		Not stated ($n = 20$)
Sucrose	1	Not stated ($n = 1$)
Egg and olive oil	1	Not stated ($n = 1$)
Not stated	39	Not stated ($n = 39$)

Information, File S3 (https://www.jfda-online.com/journal/vol31/iss2/3/). Of the studies included in this review, only 6 (8%) described all seven identified methodological processes, while more than one quarter (20; 28%) described ≤2 of these. The steps least frequently reported included: sample weight (46 studies (65%) did not report), sample presentation approach (40 studies (56%) did not report), equipment calibration method (39 studies (55%) did not report), post-dehydration homogenisation process (38 studies (54%) did not report), and sample analysis frequency (29 studies (41%) did not report). Thirty-four studies were also excluded from this review due to failing to provide sufficient methodological detail on the energy measurement processes undertaken.

3.4. Studies quantifying impact of sample process variance

Only two studies explored aspects of methodological variance and the potential impact this had on gross energy measurement (Table 6). One of these [47] compared gross energy densities of fish using three different homogenisation methods: (i) drying prior to homogenising; (ii) homogenising prior to drying; and (iii) autoclaving and homogenising prior to drying. The other study [33] compared freeze drying and oven drying of banana samples.

4. Discussion

This review evaluated sample preparation and equipment calibration processes in studies that measured gross energy content of food and beverages via bomb calorimetry. Only six [13,16,29,30,38,66] of the 71 included studies described all seven methodological processes identified for conducting bomb calorimetry. While 105 studies were initially identified as being eligible through the literature screening process, 34 studies ultimately had to be excluded from this review as they failed to provide sufficient methodological detail on the sample preparation or equipment calibration processes undertaken. Methods employed in bomb calorimetry studies of foods and beverages appear to be highly heterogenous and are often poorly described. This raises questions around the accuracy of many studies' findings, presents challenges when interpreting and comparing results between studies, and reduces the capacity to conduct reliable and repeatable research.

4.1. Sample preparation

4.1.1. Initial homogenisation

Around three quarters of studies reported an initial homogenisation process during the first phase of the food sample preparation process. Homogenisation is considered a crucial step to facilitate even distribution of the energy-derived components within the test sample [47]. Without appropriate homogenisation, samples may not be representative of the complete product; rather, analysed as a concentrated element of a product's constituents. This step is especially important for mixed samples such as meals containing several food components/ingredients, which may represent a considerable proportion of many individuals' caloric intake [63].

4.1.2. Sample dehydration

Most studies reported undertaking a liquid removal process (i.e., dehydration) during food sample preparation. This was typically done using freeze drying or oven drying techniques. Liquid removal enables combustion of dry sample matter [1], which is crucial to ensure complete combustion and energy capture. Early research has indicated that drying at temperatures of 70°C and upward may result in volatilisation of essential oils [84] and decomposition of fats and carbohydrates in foods [85]. Studies using biomass material have revealed that exposure to temperatures above 105°C for liquid removal can result in loss of volatile matter [86] and may cause degradation of unsaturated fats and caramelisation of sugars [87–89], potentially influencing combustion values. In contrast, other

Table 6. Studies reporting aspects of methodological variance.

Study	Food item	Method	Result	Outcome
Glover (2010)	Fish	1. Drying prior to homogenising 2. Homogenising prior to drying 3. Autoclaving + homogenising prior to drying	CV = 2.3% CV = 2.3% CV = 1.1%	Lower CV observed using autoclave method. This meant analysis of a third pellet was required for fewer fish (i.e., a third pellet was combusted when the first two deviated more than 2% from each other)[a]
Borah (2021)	Banana	1. Freeze drying 2. Oven drying	356.23kj/100g vs 357.17kj/100g	Oven drying provided a slightly higher combustion reading (0.26%) vs freeze drying

CV = Coefficient of variation.

[a] Method 1 = 47%, Method 2 = 60%, Method 3 = 20%.

research suggests that using different drying temperatures (i.e., 70°C in an oven vs 120°C in an autoclave) does not affect gross energy measurement [47]. At present, it remains unclear the extent to which temperature variance in the liquid removal process of food and beverages may influence subsequent sample combustion values. Given this, a prudent approach would be to complete any liquid removal employing the lowest effective temperature.

4.1.3. Post-dehydration homogenisation

Less than half of the included studies specified a post-dehydration homogenisation process. During the dehydration process, separation of product constituents may occur (e.g., lower density elements rising to the top of the sample). Similar to the initial homogenisation process, this step is vital for even re-distribution of energy-providing constituents in the combusted sample, ensuring it is representative of the actual product [1]. Despite this, most studies did not clearly indicate if, or how, this process was undertaken. Hence, the extent to which this aspect of sample preparation impacts subsequent gross energy values remains unknown.

4.1.4. Sample presentation

Less than half of the studies included in this review reported a sample presentation process prior to combustion. Of those that did, most reported samples being compressed into a pellet. Creating a pellet or encapsulating the sample creates a fuse-like environment, whereby the burning surface area is reduced, causing the sample to burn more evenly [1]. Despite bomb calorimeter manufacturers recommending gelatine encapsulation as a method for sample containment during combustion [19,20], no studies reported using this approach. This may be due to the additional steps involved in packing the sample homogenate powder inside a capsule and incorporating the relevant spike value into calculations to account for the mass and energy contribution of the capsule material. One study used sawdust as a carrying substrate and spiking agent for liquid samples (olive oil and alcohol) [45]. This process eliminated several steps, such as liquid removal and homogenisation, reducing preparation time. However, there is limited research regarding the use of this technique and further investigation is warranted to determine the potential impact of this approach on caloric determination of samples.

4.1.5. Sample weight

Only a third of the studies detailed the sample combustion weight for calorimetry analysis. An adequate amount of sample ensures sufficient combustion and an accurate capture of gross energy via temperature rise [1]. Bomb calorimeter manufacturers often indicate that the amount of sample used for calorimetry is dependent on the caloric density of the product and its constituents. For example, high fat products may combust completely with lower sample amounts, while higher carbohydrate and protein products may require a larger sample amount. Food sample amounts are generally recommended to be between ~0.3g (i.e., oil, fat) to ~0.75g (i.e., sugar) [19]. This is reasonably consistent with reports from studies included in this review (for those that indicated sample amounts), as most samples with higher carbohydrate content (i.e., mixed diet, bakery, crops) were analysed with sample amounts of 1g or more, while fish samples (i.e., high fat) were all analysed using sample amounts of 0.4g or less.

4.1.6. Sample frequency

Just over half of the studies in this review reported sample analysis frequency. In most cases, this was undertaken in duplicate or triplicate. Conducting a larger number of combustions facilitates the determination of sample variance (i.e., confidence intervals). This is important, as individual measures may be influenced by factors such as operator or machine error, or intra-sample variation (i.e., uniformity difference in samples that have not undergone sufficient homogenisation). Due to the majority of studies not reporting a post dehydration homogenisation process, sample uniformity is pertinent. Given a quarter of included studies only employed individual or exclusively duplicate sampling frequencies, in addition to 41% failing to report any analysis frequency figure, many studies fail to adequately quantify the variance of their caloric analysis.

4.1.7. Equipment calibration — method and frequency

Less than half of studies reported using an equipment calibration method. Machine calibration is an integral step to facilitate machine reporting precision and is recommended by bomb calorimeter manufacturers to occur at least daily [19,21]. The calibration process generally involves combusting a substance of known calorific value and comparing this to the standard. The International Organization for Standardization (ISO 1928) specifies combustion of certified benzoic acid as the preferred method for bomb calorimeter calibration [4]. Most studies that did report a calibration method used benzoic acid, especially those conducted more recently (i.e., post year 2000). Only eight studies listed calibration frequency, with half of these reporting machine calibration being undertaken after every ten sample combustions. Besides daily commencement calibration, manufacturers may recommend calibrating the calorimeter after a set number of combustions (e.g., every 10 burns), when changes to equipment occur (i.e., replacing O-rings, firing wire, etc.), and to account for deviations in ambient conditions (i.e., when the room temperature changes by more than 2°C) [19,21]. Given this, the current study suggests a concerning lack of reporting on calibration processes used within food/beverage bomb calorimetry research.

4.2. Sample preparation comparisons

Only two studies were identified that directly examined the impact of manipulating sample preparation techniques on the gross energy measurement of food (Table 6). One of these studies examined different initial homogenisation approaches for fish and found that a lower coefficient of variation was observed when an autoclave method was used prior to homogenisation and drying. This meant that analysis of a third sample was required on fewer occasions (i.e., when the first two pellets deviated by more than 2%) when using this method [47]. In the latter study, liquid removal processes were compared (i.e., freeze drying vs oven drying) for the analysis of banana samples, with results indicating that oven drying produced slightly higher combustion values [33]. Collectively, these findings indicate that employing different sample preparation techniques may result in subtle variation in gross energy measures. Despite this, many of the sample preparation techniques and the impact they have on energy determination have not been directly studied across a range of food/beverage products. The extent to which changes in methodological processes may influence gross energy measurement via bomb calorimetry requires further exploration.

4.3. Recommendations for reporting of bomb calorimetry studies of foods and beverages

To improve the rigor of studies using bomb calorimetry to measure the gross energy content of foods and beverages, standardised reporting of methodological techniques and processes are required. To facilitate this, we have developed the Bomb Calorimetry Methodology Reporting Checklist for Researchers, with guidance on each of the seven identified steps used in bomb calorimetry (Table 7). While developed as a reporting guideline, this checklist may also be used to assess the methodological quality of bomb calorimetry studies.

Table 7. Bomb calorimetry methodology reporting checklist for researchers.

Process	Item	Checklist Description	Details[a]
Initial sample homogenisation			
	1a	Specify homogenisation method undertaken prior to liquid removal (i.e., blended, mixed, ground, minced, cut)	
	1b	Report equipment name and type	
Sample dehydration			
	2a	Identify liquid removal process (i.e., freeze dry, oven dry)	
	2b	Specify temperature	
	2c	Specify time	
	2d	Specify if sample was dried to a constant weight	
Post-dehydration homogenisation			
	3a	Distinguish homogenisation method undertaken post liquid removal (i.e., blended, mixed, ground)	
	3b	Report equipment name and type	
Sample presentation			
	4	State final sample presentation approach (i.e., pellet, capsule, paste, raw)	
Sample weight (mass)			
	5	Report sample mass analysed (i.e., 0.5g, 0.75g, 1.0g)	
Sample frequency			
	6	Identify sample analysis frequency (i.e., single, duplicate, triplicate)	
Equipment calibration − method and frequency			
	7a	Specify bomb calorimeter calibration method (i.e., benzoic acid)	
	7b	Specify bomb calorimeter calibration frequency (i.e., daily, after every × 10 sample combustions, etc.)	
	7c	Report equipment name and type	

[a] Some methods may not be appropriate for certain food or beverage types (e.g., dehydration of olive oil).

4.4. Strengths and limitations

This study has several strengths. It is the first review to coalesce available literature describing food and beverage sample preparation processes prior to undertaking bomb calorimetry. In this review, methodological rigor was supported by following PRISMA-ScR reporting guidelines for scoping reviews [18]. The review incorporated peer reviewed literature from five major databases using broad search terms to maximise scope. The chronological age of studies ranged from the late 1960's to present, ensuring a comprehensive range of literature was sourced. Two thirds of studies were published since the year 2000, indicating most of the included studies were likely to reflect current processes. Finally, this review resulted in the development of a reporting checklist for bomb calorimetry studies which has the potential to improve methodological documentation and hence quality of future bomb calorimetry research.

This study also has some limitations. First, only studies published in English were included in the review. This resulted in the omission of at least one study (Portuguese). Second, we did not apply our search strategy to all academic databases; only those indicated in Supporting Information_2. Databases were selected based on their comparable research fields and applicable interest areas and were considered most appropriate given the scope of the review. Nonetheless, this limits inclusion of grey literature such as food industry reports, which may employ high standards for calorimetry processes and reporting. We did however consult bomb calorimeter manufacturer operations manuals, including those developed by the Parr Instrument Company, which made up the majority of calorimetry machines used in studies within this review [19,20].

4.5. Conclusions

This review provides an evaluation of the sample preparation and calibration techniques used for measuring the gross energy content of food and beverages via bomb calorimetry. Overall, this review highlights that a variety of techniques are employed to prepare food and beverage samples for combustion, and these are not always well described. The extent to which these methodological variations may impact gross energy determination is currently unclear, potentially reducing confidence in study findings. This also has implications for the replicability of research and may preclude direct comparisons being made between studies. Further research is needed to examine whether the identified sample preparation techniques and bomb calorimetry measurement processes are appropriate, and to quantify the impact of methodological heterogeneity on gross energy values. The development of a Bomb Calorimetry Methodology Reporting Checklist for Researchers presents an opportunity to navigate some of these issues. The information in this review may help guide future food and beverage sample preparation processes, in turn improving the accuracy and efficiency of gross energy determination. It may also assist with the development of clearly defined standards outlining the methodological processes required for the conduct of bomb calorimetry with food and beverages.

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Author contributions

ZH conducted the literature search; extracted, collated, analysed, and interpreted the data; and drafted the manuscript. BD, CI and SR supervised this process and critically reviewed and revised the manuscript. All authors contributed to the conceptualisation and design of this review and the study selection; and have read and approved the final version submitted for publication.

Transparency declaration

The authors affirm that this manuscript is an honest, accurate, and transparent account of the study being reported. The reporting of this work is compliant with PRISMA guidelines/AMSTAR checklist. The authors affirm that no important aspects of the study have been omitted and that any discrepancies from the study as planned (OSF Registration DOI https://doi.org/10.17605/OSF.IO/JFHDT) have been described.

Conflict of interest

The authors have no conflicts of interest.

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