

Article

Self-Heating Risk of Coals and Metal Powders: A Comparison

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Abstract: The self-heating risk of four coal samples (two bituminous and two subbituminous) and five metal powder samples (four industrial and one laboratory-prepared) were studied calorimetrically using oxidation heat at 30 °C. All of the samples were measured in fresh (as-received), vacuum-dried and wetted states. The heat effects of fresh and/or dried coals were found to be significantly higher than those of the metals. On the other hand, wetting the samples markedly increased the oxidation heat mainly of the metals, making their oxidation potential comparable to or even exceeding that of the subbituminous coals. As a practical consequence, a comprehensive index to assess the self-heating risk of the materials in respect to both their oxidation ability and the effect of moisture is proposed.

Keywords: metal powder; coal; self-heating risk; oxidation heat; calorimetry

1. Introduction

The phenomenon of self-heating is often related to coal and coal products [1,2]. With the development of the industry, however, the role of metal powders has significantly grown, with non-carbonaceous materials having also been confirmed as susceptible to the self-heating process [3–8]. Among the metals prone to self-heating oxidation aluminium, iron, tin, uranium and/or zinc can be mentioned [3,4,6]. According to USA statistics, over the period of 1980–2005, 24% of accidents due to self-heating, fire and/or explosions were found to be related to metal dust, while only 10% of them were caused by coal [9]. An inherent requirement of industrial producers and users is assessing the self-heating potential of substances while keeping the basic influencing factors (temperature, grain size, moisture content, etc.) constant and/or under control during evaluations. In principle, there is still no indicator to assess the risk of self-heating simultaneously for both coals and metals. As coal is concerned, a number of different methods have been proposed (e.g., [10,11]), among which calorimetry is of exclusive importance. The main argument for calorimetry is the possibility to directly measure the oxidation heat, which is considered the main cause of self-heating occurrences [12–14]. As for metal powders, DSC investigations are often preferred, however, enabling investigations of the oxidation behaviour of metals mainly at elevated temperatures [3,15]. Calorimetric studies of metals' oxidation at ambient temperatures are rather scarce [16–18].

This paper aims to compare the self-heating risk of coal and metal powder samples using directly measured oxidation heat at 30 °C. Wetting the samples is demonstrated to markedly increase the oxidation heat mainly of the metals, making their oxidation potential comparable to or even exceeding that of the subbituminous coals. As a result, a complex index reflecting the self-heating risk of materials both in dry and corrosive/wet surroundings is suggested.

2. Materials and Methods

2.1. Samples

Four samples of coal (two bituminous and two subbituminous) and five metal powder samples (four industrial and one laboratory-prepared) were studied.



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For the coal representative samples, two bituminous coal types (denoted as B1 and B2) from the Ostrava-Karviná Coal Basin and two subbituminous types (denoted as SB1 and SB2) of the North Bohemian Coal Basin were studied.

For the metal powders, four samples (Al₁, Al₂, Mg and Sn/Zn) were obtained from industrial enterprises, and one sample (Fe) was prepared in laboratory. The industrial samples were collected from containers used for waste dust (samples Al₁, Mg and Sn/Zn) or from filtration unit of a cleaner (sample Al₂). The Fe sample was prepared in the laboratory by grinding an iron cylinder using a rotary grinder, with the iron particles being collected in a vessel filled with liquid nitrogen. This setup was used to prevent the Fe particles from oxidizing.

2.2. Oxidation Heat Measurements

To determine oxidation heat, C80 calorimeter (Setaram, Lyon, France) was used following the method of pulse flow calorimetry [19]. Measurements were taken at basic temperature of 30 °C and pressure about 100 kPa. A discrete amount (pulse) of oxygen was introduced into the flow of inert gas (helium), and the heat of chemical (irreversible) reaction between oxygen and the investigated sample was evaluated. The flow rate of helium was about 2.5 mL min^{−1}. The volume of the oxygen pulse was 80 mL, and, thus, the passage of oxygen through the sample took about half an hour. The mass of the sample was about 2 g. Typical pulse flow calorimetric curve (as obtained of the measurement of coal sample B1) is depicted in Figure 1.

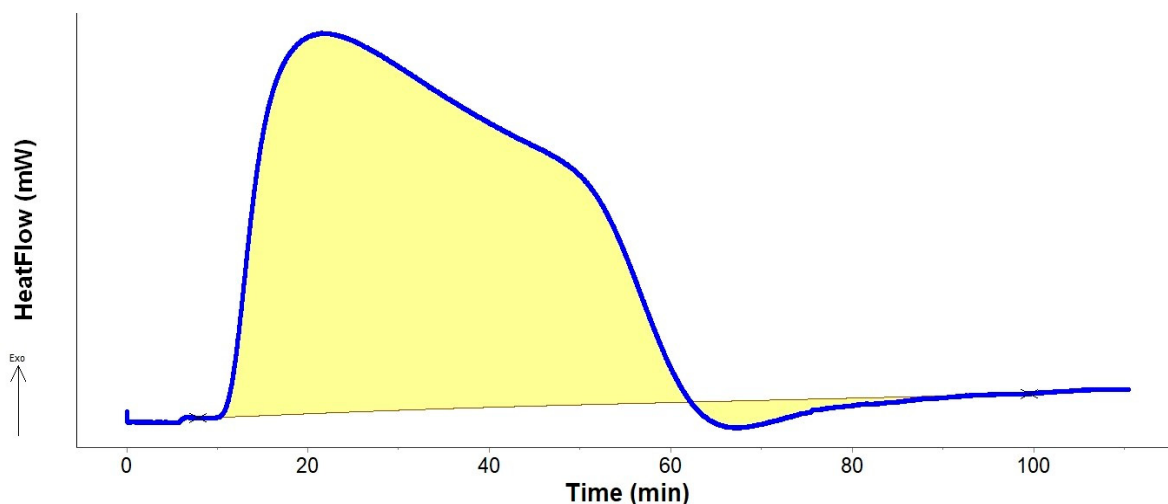


Figure 1. Pulse flow calorimetric curve of sample B1—original state. Area above the baseline reflects exothermic effects of irreversible/chemical and reversible/physical interactions of oxygen with sample. Area below baseline is proportional to reversible interaction only [19].

From the calorimetric tests, the mean oxidation heat flow q_{30} during the 30 min reaction was determined ($\text{J (kg} \cdot \text{s)}^{-1}$, equivalent to W kg^{-1}), with the index of 30 indicating temperature level and the residence time of oxidation. The standard deviation for the oxidation heat is about 7% and was determined from five replicated measurements of the SB2 sample (dried state) and the Al₁ sample (wetted state).

2.3. Pre-Treatment of the Samples

The samples were calorimetrically measured in fresh (as-received), vacuum-dried and wetted states, respectively.

As-received/fresh state: In fresh state, coal samples were measured. Specifically, coal lumps were taken from the coal seam and immediately sealed in an airtight plastic bag. Just before the calorimetric measurements in laboratory, the coal lumps were crushed, milled,

and sieved to achieve a uniform grain size of 0.06–0.15 mm. The crushing and sieving of coals were carried out in air as fast as possible, and it took less than 30 min before the sample was put into the flow of the inert gas in calorimeter.

Metal powders were taken from the above-mentioned containers/filtration unit of given enterprises and placed into airtight bags, then transported to laboratory and stored in a refrigerator until they were measured. However, the powders were not specially prevented from air before their taking from the containers, and, uncontrolled oxidation of their surface could occur. All the industrial metal samples can thus hardly be considered fresh, and they are denoted “as received”. An exception is iron sample (Fe), which was prepared in laboratory using rotary grinder with the evolved particles caught in liquid nitrogen. It was only in contact with aerial oxygen for a few seconds before calorimetric measurement took place, so the sample is regarded as fresh.

Dried state: Each sample was dried under vacuum (90 °C for ca. 1 h). Before drying, the oven was repeatedly evacuated and flushed with nitrogen. When dried, samples were cooled under nitrogen atmosphere to room temperature and immediately measured with the calorimeter.

Wetted state: The samples tested previously in dried state were subsequently exposed to wetting procedure with the aim to make the samples moist and thus illustrate the effect of moisture on the oxidation process. Therefore, the samples were shortly (for about 1–2 min) exposed to the flow of water vapour generated by boiling water. After the wetting treatment, it took less than about 5 min to insert the wetted sample into the inert flow in calorimeter. The moisture content of the samples was determined after the calorimetric test (drying in air at 105 °C for 2 h).

2.4. Chemical Characterisation

Coal samples are here characterised using carbon content on dry, ash-free basis: C^{daf} (%). For the analyses, standard procedure using LECO elemental analyser was applied.

For chemical characterisation of metals, samples were dissolved in a mixture of hydrochloric and nitric acids and analysed for metals using atomic absorption spectroscopy (AA240FS, Varian, Palo Alto, CA, USA). The content of SiO_2 was determined gravimetrically as insoluble residue after the sample dissolution. The amount of combustibles was evaluated as decrease in mass at 550 °C from thermoanalytical investigations in air atmosphere (Setaram Setsys Evolution, Lyon, France).

2.5. Grain Size Measurements

The grain size distribution of metals was determined using dispersion unit of Mastersizer 2000 analyser (Malvern, Great Britain) equipped with SW V5.60. Ten replications were conducted for each sample. Particle size distribution curves of the metals are illustrated in Figure 2.

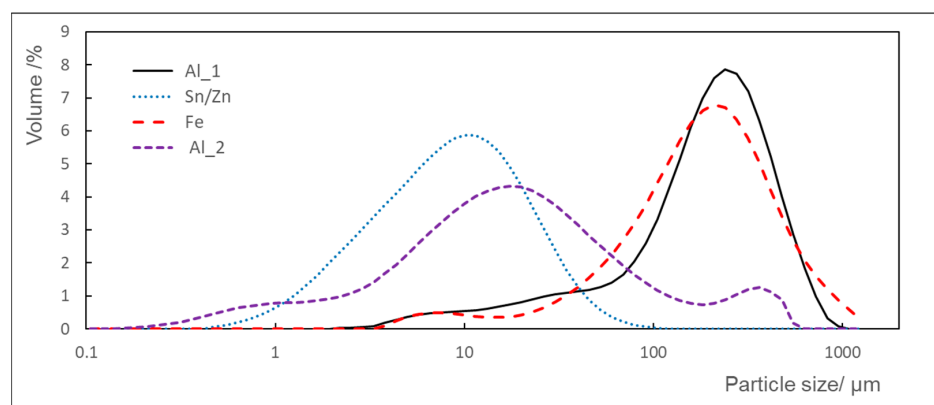


Figure 2. Particle size distribution curves of metal samples. (The peak of the curve represents the most abundant particle fraction for each given sample).

The numerical results of the measurements are represented using nominal diameter of the particles: d_{50} (μm).

3. Results and Discussion

3.1. Texture of the Samples

The contents of the main elements in the individual samples are shown in Table 1. Based on the analysis, the prevailing content of the principal element can be seen for each sample, except for sample Al_2, which contained slightly more than 50% aluminium, thus confirming the contamination of this sample due to workplace impurities. Among the contaminants, silica gel (19%) and a large amount of combustibles (26%) were mainly identified. In the series of samples, the mixture sample of Sn/Zn is also atypical, showing a roughly equivalent content of both main elements.

Table 1. Comparison of oxidation behaviour of coals and metal powders; W —moisture content of sample at the time of measurement; d_{50} —nominal diameter of particles; d_{af} —dry, ash-free basis.

Sample	Description (Technology Type)	Grain Size/ d_{50} (μm)	Content of Main Element (%)	Original State		Dried State		Wetted State	
				W (%)	q_{30} ($W\text{ kg}^{-1}$)	W (%)	q_{30} ($W\text{ kg}^{-1}$)	W (%)	q_{30} ($W\text{ kg}^{-1}$)
Coals									
B1	Bituminous	60–150	$C^{\text{daf}} = 87.6$	1.5	0.52 **	<0.1	0.25	3.6	0.30
B2	Bituminous	60–150	$C^{\text{daf}} = 92.5$	1.1	0.36 **	<0.1	0.18	2.5	0.25
SB1	Subbituminous	60–150	$C^{\text{daf}} = 72.6$	25.4	4.1 **	<0.2	1.5	14	3.0
SB2	Subbituminous	60–150	$C^{\text{daf}} = 71.3$	28.4	3.0 **	<0.5	2.3	17	3.9
Metals									
Al_1	Aluminium (grinding)	190	Al = 85	0.4	0.12 *	~0	0.011	2.8	11.7
Al_2	Aluminium (welding)	16	Al = 53	2.3	0.083 *	~0	0.040	13	2.8
Mg	Magnesium (cutting)	-	Mg = 95	0.6	0.045 *	~0	~ 0.01	2.0	0.32
Sn/Zn	Sn-Zn (metal coating)	11	Sn = 50; Zn = 44	0.8	0.083 *	~0	0.043	7	1.3
Fe	Iron (grinding-lab)	170	Fe = 93	0.8	0.47 **	~0	0.15	19	31.0

**—fresh state; *—as received state.

The coals were investigated in a uniform grain size interval of 60–150 μm , while the granularity of the metal samples was different. The coarsest samples are the Al_1 and Fe samples, with nominal diameters of d_{50} 190 μm and 170 μm , respectively. In contrast, sample Al_2 and the mixture sample of Sn/Zn consist of the finest particles, with nominal diameters of d_{50} 16 μm and 11 μm , respectively.

3.2. Oxidation Heat

The results of the calorimetric investigations are summarised in Table 1.

For the original state of the samples, the highest oxyreactivity is clearly exhibited by the fresh subbituminous coals, with oxidation heat values an order of magnitude higher than the q_{30} values of the other samples. In contrast, the industrial metals displayed the lowest oxidation heat values, primarily due to the uncontrolled passivation of the metal surface by atmospheric oxygen during prior industrial processing and storage. Though generally finer particles react more intensively with oxygen [3,4,6,8], the degree of the surface passivation outweighs the effect of grain size. This is evident in the case of the aluminium samples: sample Al_1 with a nominal diameter of 190 μm showed a higher level of oxidation heat than the fine-grained aluminium Al_2 sample, with a d_{50} of 16 μm (see Table 1). As a result, the q_{30} values measured for the industrial metals reflect their residual oxyreactivity rather than their original oxyreactivity.

The calorimetric technique can be used to evaluate the self-heating propensity of samples [12–14]. However, to reliably compare the self-heating risk of different specimens,

it is essential that the samples are measured when they are in well-defined and comparable states. For coals, the fresh state is commonly used as the basis for comparison [20]. Using the method of pulse flow calorimetry, the following boundaries for self-heating risk categories depending on coal rank can be applied [21]: subbituminous coals: $q_{30} > 4 \text{ W kg}^{-1}$ —high risk, $q_{30} < 2 \text{ W kg}^{-1}$ —low risk, $2\text{--}4 \text{ W kg}^{-1}$ —medium risk; bituminous coals: $q_{30} > 0.45 \text{ W kg}^{-1}$ —high risk, $q_{30} < 0.2 \text{ W kg}^{-1}$ —low risk, and $0.2\text{--}0.45 \text{ W kg}^{-1}$ —medium risk. Using the classification scale, coals SB1 and B1 can thus be ranked as samples with a high self-heating risk, while samples B2 and SB2 belong to the category of medium-risk samples. Unfortunately, similar boundaries cannot be proposed for industrial metals due to uncertainties in their pre-oxidation history—thus, we could not ensure a comparable/fresh basis during the measurements. The effect of the freshness of the metal surface on its oxyre-activity can be demonstrated by the Fe sample that was prepared in the laboratory. The fresh surface of the Fe particles had an oxidation heat about four to ten times higher than the values of q_{30} for the industrial samples in the “as received” state (cf., Table 1). Moreover, the coarse character of the Fe sample (with a nominal diameter $d_{50} = 170 \text{ }\mu\text{m}$) indicates the value of q_{30} for the Fe sample would evidently increase if it was obtained with a finer character. Irrespective of the coarse grain size of the Fe sample, its oxidation heat q_{30} would place it within the high-risk category according to the scale valid for bituminous coals.

The calorimetric results in Table 1 then clearly demonstrate that the drying pre-treatment caused a decrease in oxyreactivity for both the coals and metals. Thus, the level of oxidation heat q_{30} was roughly half that obtained for the original state. In the dried state, the subbituminous coals again showed the highest oxyreactivity of all the samples.

The behaviour of the samples towards oxygen was fundamentally altered by the wetting procedure. In principle, the oxidation heat of all the moist samples increased. However, while the change in oxyreactivity of the wetted bituminous coals was minimal (if any), the oxidation heats of the moist subbituminous coals increased by 60–100%. This significant increase was found in both the as-received industrial metal samples and the fresh Fe sample. Moreover, the synergistic effect of water appears to be largely independent of the particle size of the metals within the studied range of $d_{50} \sim 10\text{--}200 \text{ }\mu\text{m}$ (cf., Table 1). As a result, the moist metals exhibited oxyreactivity comparable to or even exceeding that of the subbituminous coals (cf., samples Al_2, Al_1 and Fe; Table 1).

The accelerating effect of moisture can be explained by the involvement of water in the mechanistic pathway of the oxidation process. The active role of water has already been described for subbituminous/hydrophilic coals [19,22,23] as well as for metal powders [5,17].

In the case of coals, the formation of hydroperoxides is likely accelerated in the presence of water, with this reaction assumed to be a key step in the mechanism of coal oxidation at low temperatures [24–26].

For metals, the alteration in the oxidation mechanism then involves a greater diversity of oxidation products. Specifically, instead of the formation of “pure” metal oxides (Me_xO_y), which is typical for “dry” oxidation [3,17,27], the presence of water leads to the production of a mixed oxide–hydroxide phase $\text{MeO}_x(\text{OH})_y$, which is energetically more favourable [5,18,28]. Additionally, the involvement of the electrochemical aspects in the oxidation mechanism of the wet metal surface appears to play an important role [5,28].

Repeated wetting experiments allowed for the characterisation of the effect of water on oxidation heat over a broad range of moisture contents W . For the subbituminous coal SB1, the dependence of heat q_{30} on W is shown in Figure 3. The maximum value of oxidation heat q_{30} at a moisture content of about 15–20% is evident, indicating the optimal conditions for the involvement of water in the oxidation mechanism [12,22]. Moisture contents exceeding this “optimal” value apparently hinder the access of oxygen to reaction sites on the surface, as deduced from the lower values of q_{30} for the very moist samples.

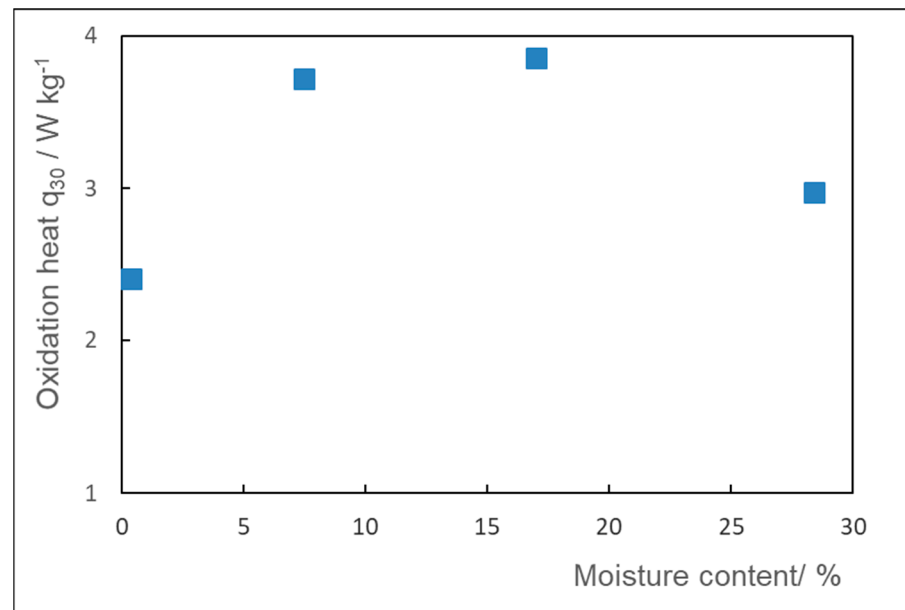


Figure 3. Oxidation heats q_{30} of sample SB1 as a function of moisture content, W .

The repeated wetting procedure for the metal samples differed. Specifically, five replications of the wetting experiments with sample Al_1 resulted in a relatively constant moisture increase of only about 2.3–3.3%, leading to a mean oxidation heat value of $q_{30} = 11.7 \pm 0.7\ W\ kg^{-1}$ (cf., Table 1). This observation is likely due to the hydrophobicity of the metal surface [18]. To study the effect of moisture content on the oxidation of the Fe sample, a highly wetted state was thus achieved by adding an appropriate amount of liquid water to the dry sample. This method allowed for the oxidation heat q_{30} to be measured across a broad range of moisture contents for the Fe sample, as shown in Figure 4.

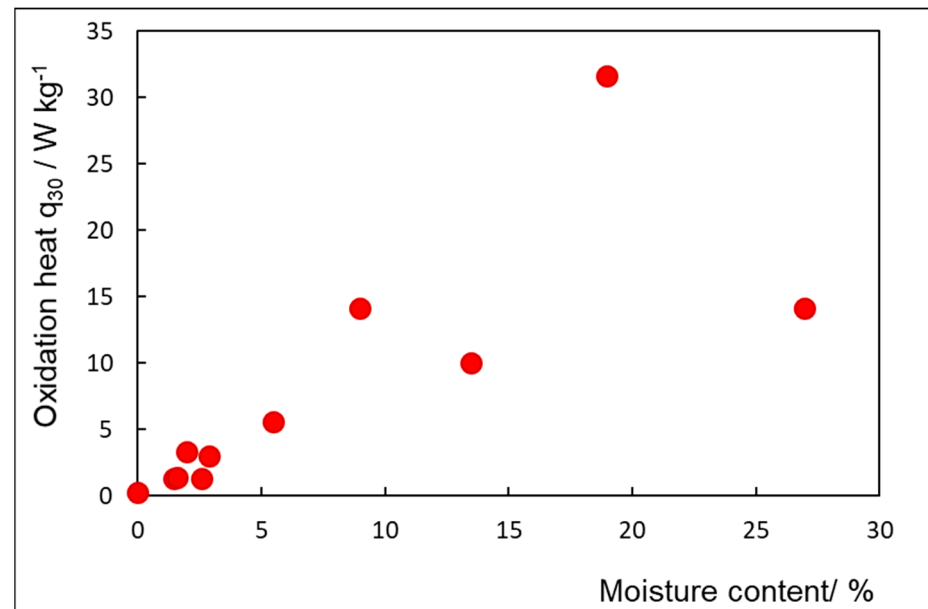


Figure 4. Oxidation heat q_{30} of the Fe sample by moisture content.

The dependencies shown in Figures 3 and 4 are qualitatively similar, with both exhibiting a peak in oxidation heat at a moisture content of about 15–20%. However, the actual values of oxidation heat at the peak differ significantly. Specifically, the maximum q_{30} value for subbituminous coal is approximately $4\ W\ kg^{-1}$, representing about a twofold

increase compared to the q_{30} value in the dry state. In contrast, the maximum q_{30} value for the Fe sample exceeds 30 W kg^{-1} , which represents an increase of more than two orders of magnitude compared to the oxidation heat of the dried Fe sample. In fact, such a high oxidation heat value as observed for the Fe sample at its peak was never matched by the coal samples in our laboratory [21].

3.3. Consideration of Comprehensive Self-Heating Risk Indicator

Based on the investigation of the oxidative behaviour of coal and metal samples, a new indicator (q_{30}^W) is proposed to assess the self-heating risk of these materials. The q_{30}^W indicator accounts for oxyreactivity and the effect of water, distinguishing the self-heating risk in dry versus wet/corrosive environments. It is derived from the oxidation heat values measured in both the dried and wetted states of the samples. The effect of moisture on the oxidation process is quantitatively expressed as the ratio of the oxidation heat of the wet samples to that of the dried samples (R). Numerically, the q_{30}^W indicator consists of the oxidation heat value q_{30} of the dry sample, followed by the ratio R , with the moisture content W of the wet sample noted in parentheses. Using this approach, the q_{30}^W indicator for the coal sample SB1 (Table 1) would be represented as $1.5 \text{ W kg}^{-1}/2$ (14%), for example.

Thus, the criterion for assessing the self-heating risk of a sample is its oxidation heat value in the dry state q_{30} , along with the ratio R , which reflects the effect of moisture. A high q_{30} value, regardless of the R ratio, classifies the sample as having a high self-heating risk in both wet and dry environments. Conversely, a low q_{30} value coupled with a high R ratio indicates a low degree of self-heating risk in dry conditions but a high degree of risk in wet environments. However, determining precise threshold values for categorizing different levels of risk is a matter to be explored in further investigations.

4. Conclusions

The obtained results underline the crucial importance of sample history and moisture content in the investigation of the oxidation heat of coals and metal powders. Water was observed to have an enormous effect mainly on metals, with the oxidation heat rising by up to two orders of magnitude. Sample history and the effect of water are separately evaluated using the q_{30}^W indicator, which has been newly designed for these materials. As a result, the q_{30}^W indicator can distinguish the self-heating risk of coal and metals in dry and wet (corrosive) environments.

Supplementary Materials: The supporting information on experimental data can be downloaded at: <https://www.mdpi.com/article/10.3390/fire7110378/s1> (Calorimetric Data.xlsx).

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