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The Limiting Content of Combustibles to Prevent Minestone from the Spreading of Fire

Boleslav Taraba [1,*], Petr Gřunděl [2] and Gabriela Zelenková [1]

[1] Faculty of Science, University of Ostrava, 30. dubna 22, 701 03 Ostrava, Czech Republic; gabriela.zelenkova@osu.cz
[2] Ostravská těžební, a.s., Sládkova 1920/14, 702 00 Ostrava, Czech Republic; grundel@ostravskatezebni.cz
* Correspondence: boleslav.taraba@osu.cz; Tel.: +420-597092199

Abstract: The limiting content of combustibles to spread/suppress the fire in the minestone of the coal tailing dump (gangue) was studied. This knowledge appears crucial mainly when deciding on the possible usage of minestone as fireproof material for engineering purposes. Theoretical analysis, laboratory experiments as well as scale (in situ) considerations were performed. In the laboratory, a model series of coal–mineral matter mixtures and six representative minestone samples of coal tailing dump (gangue) were investigated. The thermoanalytical (TG/DSC) method was used to evaluate the content of combustibles with their energetic equivalent, *EEC* (%). The *EEC* has been suggested as a proper way to quantify the content of combustibles in the samples. Based on the original combustion calorimetric test, an *EEC* value of 7–9% was found to be a limit between fireproof and fire spreading minestone in a laboratory, while only 2% of combustibles resulted as the limit from the theoretical analysis. On the other hand, respecting real conditions of thermally active dump (Heřmanice tailing coal dump), the laboratory limit of 7–9% of EEC was then assessed to shift to the value of about $10 \pm 1\%$ for practice.

Keywords: coal waste dump; combustible matter; underground fire; calorimetry

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1. Introduction

Mine waste dumps are landscape formations of mined areas found in many countries around the world [1–5]. These sites are a source of rather negative environmental aspects, such as the evolution of gases/dust and the pollution of groundwater by toxic compounds [2,3,6–8]. Spontaneous thermal activity, e.g., underground fire, is one of the most dangerous phenomena in the dumps. The spontaneous heating process of coal residues in the dump is often mentioned as a cause of fires [4,5,7,9]. However, exogenic ignition, for example, from thunderstorms, lightning and/or the deposition of hot cinders, has also been reported as fire starters [6,9]. A range of papers present methods for objective detection/monitoring of underground fires [5–8], appraisal of the spontaneous heating susceptibility of the dumped tailings [4,10,11] and/or modelling the temperature field in the location with thermal activity [6,12]. The role of the oxygen with access to the firing space is often mentioned as a main factor proliferating the extent of the firing spot; on the other hand, much less attention is given to the content of combustible matters in the minestone that is limited to spreading/suppressing the fire within the dump. This knowledge appears crucial, mainly when deciding on the possible usage of minestone as a fireproof material to model the shape of tailings dumps or for engineering purposes. To our best knowledge, scarce information is mentioned only by Skarzynska in the complex study [9] designating a calorific value of 7000 kJ/kg as the threshold for the inflammability of minestone. However, a closer insight into the question is still missing.

This paper attempts to provide comprehensive information on the limiting content for combustibles in minestone, representing the boundary between possible propagation or suppression of the fire within the firing dump. The presented findings are based on theoretical analysis, original laboratory calorimetric tests as well as on scale (in situ) considerations.

2. Materials and Methods

All samples reported in the paper were taken from Heřmanice tailing coal dump, the largest one in the Ostrava-Karviná Coal District, Czech Republic. From the geological point of view, this area is the Czech part of the Upper Silesian Coal Basin. Originally, in the middle of the 19th century, the gangue was set up as a tailing for the Heřmanice Coal Mine extracting high-quality bituminous coal. For the last 30 years, underground thermal activity has been in progress there [8]. In the study, six representative samples of minestone with different content of combustion matter were used. The displayed range of 10 to 30% of combustibles practically covers the entire range of combustibles occurring in minestones deposited at the Heřmanice tailing coal dump.

Furthermore, lumps of coal proper (denoted as C) and a mudstone as "pure rock" (denoted as R) were taken from the dump to prepare a modelled series of coal–mineral matter mixtures with a defined content of combustibles/coal. For this purpose, the coal and mudstone were milled to a size below 0.2 mm. Then, a mudstone sample was heated at 900 °C for 2 h in the air to eliminate possible combustibles and stabilize the composition of the mineral matter. Subsequently, it was mixed with the coal fraction to obtain mixture series containing 5, 7, 10, 15, 20, and 30% of coal, respectively. The modelled series was used for calorimetric investigations of the limiting content of combustibles. The sample of (pure) coal "C" contained 10.4% of ash (dry basis), and it had a carbon content = 80.4% (daf basis), net calorific value = 24,500 kJ kg^{-1} (daf basis).

2.1. Method of Thermal Analysis

TG/DSC analysis was used to recognise the combustion dynamics of minestone and evaluate the content of combustible matter in the samples [13]. The measurements were performed using a simultaneous TG/DSC analyser (SetsysEvolution, Setaram, Lyon, France) coupled with a mass spectrometer QMG 700 (Pfeiffer, Wetzlar, Germany) to monitor evolved gases. For the experiments, a crucible from α-Al_2O_3 was applied with 30–50 mg of the sample under air flowing at a rate of 20 mL min^{-1}. The MS signals corresponding to H_2O (m/z = 18) and CO_2 (m/z = 44) were recorded in the multiple ion detection mode.

The combustion/burning of the minestone was studied within the temperatures 30–850 °C using a heating rate of 10 °C min^{-1}. To determine the combustibles, the sample temperature was continuously increased to 550 °C followed by the isothermal step for 1 h. The combustibles content was derived from the thermogravimetric (TG) curve as the value of the loss on ignition (LOI_{TG}) computed from the mass decrease related to the dried sample mass. An alternative procedure to determine the energetic effect of combustibles consisted of quantification of the calorimetric curve (DSC, see Section 3.1).

2.2. Combustion Calorimetry

An original experimental approach using the adiabatic combustion calorimeter C 4000 (IKA, Stauffen, Germany) was used to assess the limiting content of combustibles in minestone for the propagation/suppression of the fire under laboratory conditions. For this purpose, a series of coal–mineral matter mixtures with a defined content of combustibles were used. Two grams of the mixture were placed on a ceramic plate in the calorimeter. Then, a cotton fuse tied to an ignition wire was inserted at the margin of the sample to initiate the combustion process (see Figure 1), and the calorimetric bomb was filled with oxygen to a pressure of 3 MPa. Basic/initial temperature of the combustion experiments was 25 °C.

Figure 1. Experimental arrangement of the combustion calorimetry test to assess the limiting content of combustibles.

After the test, the character of the sample to be burned was inspected and evaluated according to the completeness of the combustion, see Section 3.2.2 below.

3. Results and Discussion

3.1. Combustibles in Minestone

The content of combustible matter in sediments is frequently estimated gravimetrically using the value of a loss on ignition, with a temperature of 550 °C being a generally accepted standard for the test [14,15]. It is assumed that the level of 550 °C is high enough for the complete combustion of all organics but, simultaneously, too low for the decomposition of carbonates. However, temperatures around 500 °C were recognised as sufficient to release interstitial water from clay admixture, making information on combustibles based on loss on ignition somewhat unreliable. Performed thermoanalytical measurements confirmed that clay minerals are also in question for the samples from the Heřmanice coal tailing dump. The situation is illustrated in Figures 2 and 3.

From the TG (black, solid) curve in Figure 2, it is evident that the sample in the air atmosphere is almost completely burned after reaching the final temperature of 550 °C (TG/DSC experimental curves for other samples are in graphical form summarized in Appendix A, Figure A1 or, in tabulated form, in Supplementary Material Sheet S1). The course of the TG curve then becomes steady, and, from the difference between the mass of the dried sample and that of the combusted sample, one can thus calculate the value of a loss on ignition, LOI_{TG}, for the given sample. The combustion is accompanied by marked heat evolution as evidenced by a large exothermic peak on the calorimetric curve, DSC (dashed, Figure 2), and by the noticeable evolution of carbon dioxide and water (see Figure 2, lower part), which are the main gaseous products of the oxidation process. However, thermoanalytical investigation of the same sample under an inert atmosphere (argon) proved the evolution of water at 550 °C also for anaerobic conditions (see Figure 3, lower part). Evidently, dehydration of the inorganic/clay matter in the sample thus occurs as also confirmed by a decrease in the sample mass and by an obvious endothermic peak (Figure 3). Therefore, the correctness of the calculated LOI_{TG} value for the real content of combustibles is undoubtedly affected.

As a solution, instead of (uncertain) gravimetric values of LOI_{TG}, the energetic equivalent of combustibles, *EEC*, is proposed here. More specifically, the heat of oxidation during combustion of the combustibles in minestone at 550 °C (Q_{net}(sample)) was measured and related to the net calorific value of pure coal (Q_{net}(coal)) taken as a comparison basis:

$$EEC\ (\%) = 100 \cdot Q_{net}(\text{sample})/Q_{net}(\text{coal}) \quad (1)$$

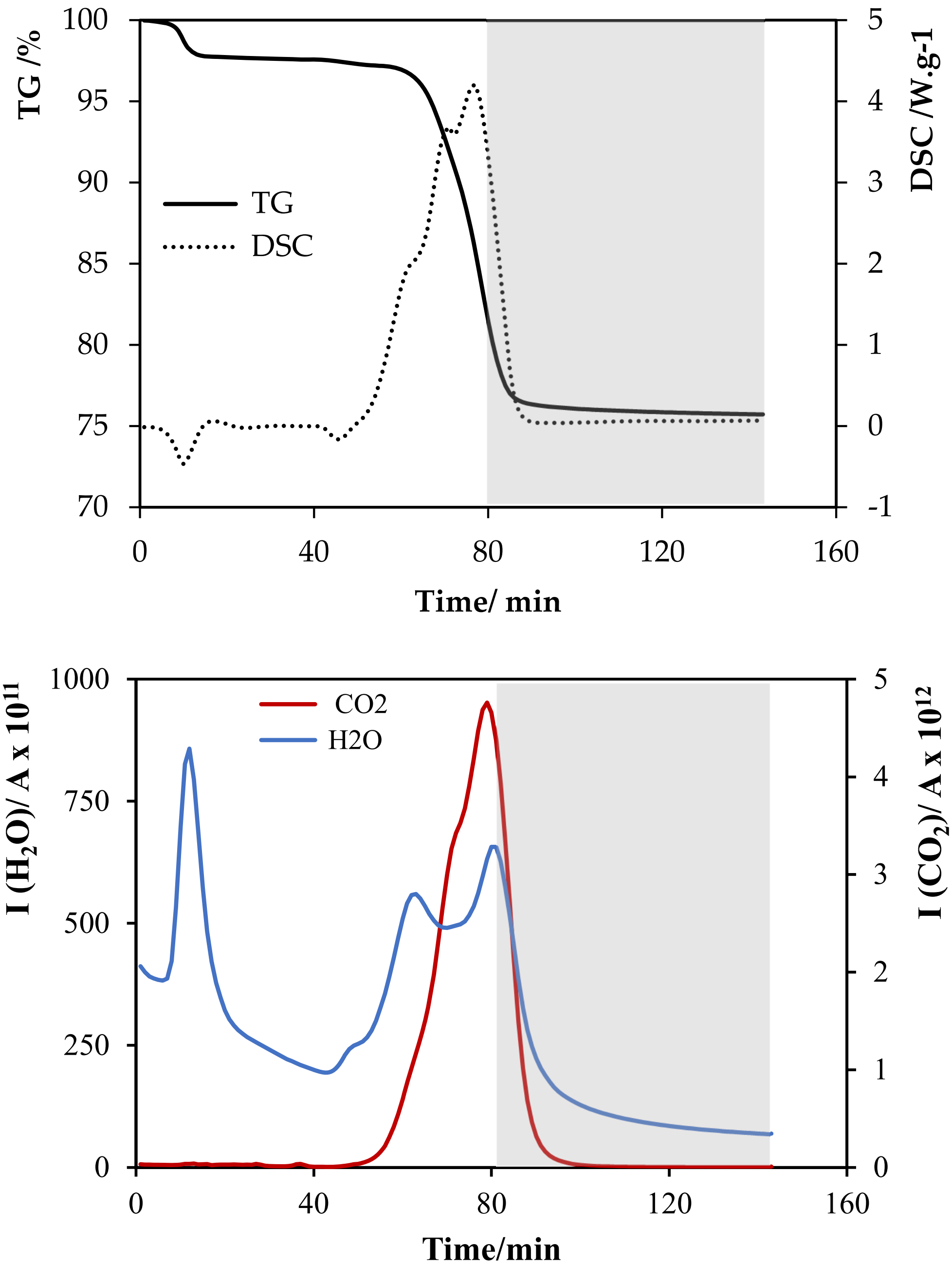


Figure 2. TG/DSC-MS curves of minestone exposed to temperatures 30–550 °C under a flow of air (sample E); the grey area denotes elapsing time with a constant temperature of 550 °C.

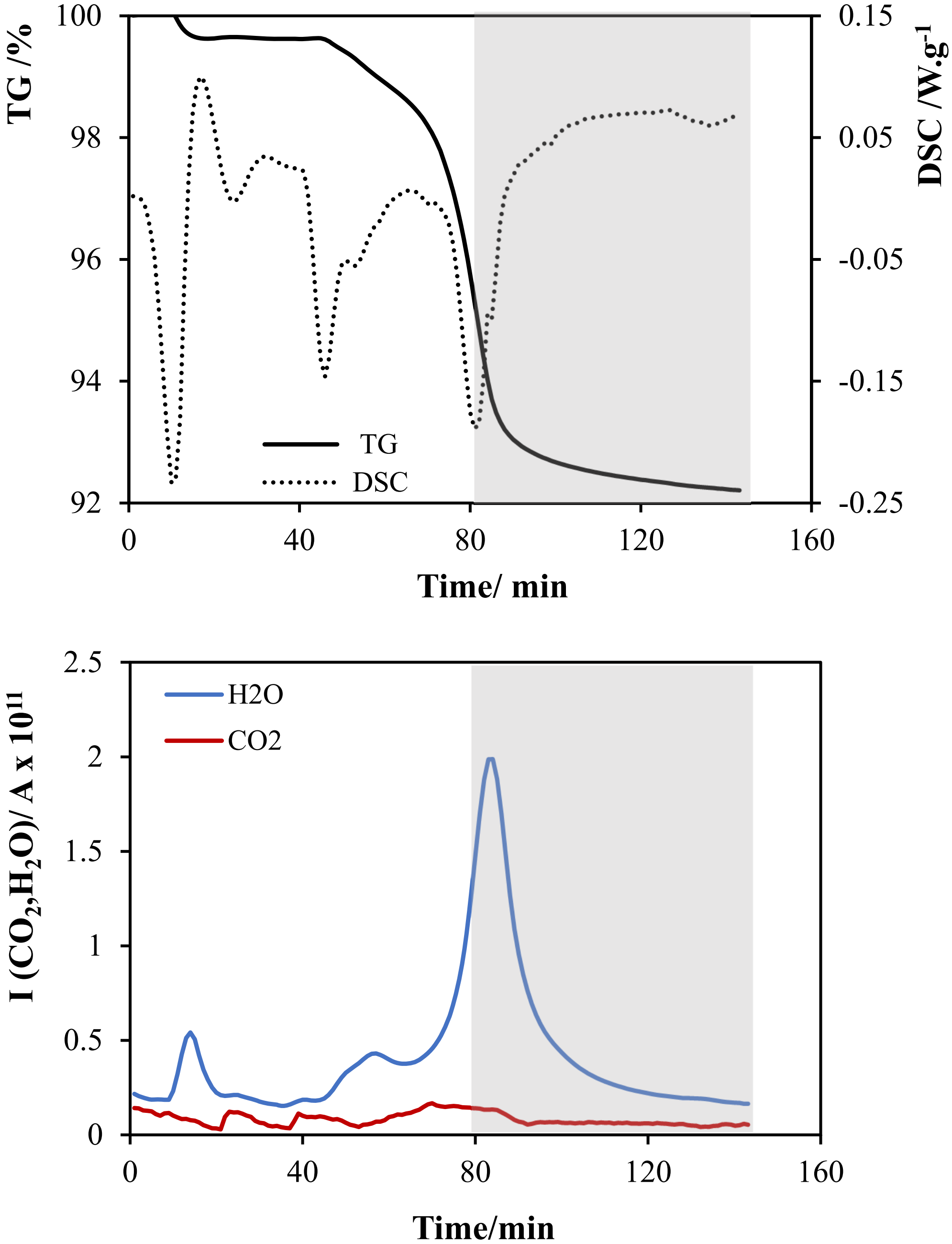


Figure 3. TG/DSC-MS curves of minestone exposed to temperatures 30–550 °C under inert (argon) atmosphere (sample E); the grey area denotes elapsing time with a constant temperature of 550 °C.

Such *EEC* values were determined for minestone samples from the Heřmanice coal tailing dump, the oxidation heat was evaluated using calorimetric (DSC) curves (cf. Figure 2), and the net calorific value of coal C taken as a standard (Q_{net}(coal) = 24,500 kJ kg^{-1}). The *EEC* values are presented in Table 1, where values of loss on ignition are also displayed as obtained from both the thermogravimetric curve (LOI_{TG}) as well from the standard gravimetric test using the muffle furnace at 550 °C (LOI_{muffle}) [5].

Table 1. Contents of combustibles using values of loss on ignition (*LOI*) and energetic equivalent (*EEC*) at 550 °C (reported values of LOI_{muffle} represent mean values of minimally double replications, experimental error for LOI_{TG} and *EEC* values is estimated as 5%rel.).

Sample	LOI_{muffle} **(%)**	LOI_{TG} **(%)**	***EEC*** **(%)**
A	10.0	10.0	7.4
B	12.8	12.1	8.5
D	17.2	16.1	16.5
E	21.3	20.6	19
F	25.1	24.1	20.8
G	27.8	28.3	26

Somewhat lower values of *EEC* compared with values of loss on ignition (roughly by 2%) can generally be deduced from Table 1. The presence of crystalline water-containing constituents in the studied samples appears to be the main reason for the difference.

3.2. The Limiting Content of Combustibles to Prevent the Spreading of Fire

3.2.1. Theoretical Approach

The main information on the behaviour of minestone, when heated to open fire, was obtained from thermoanalytical (TG/DSC) investigations under air at temperatures 30–850 °C. Typical TG/DSC development as obtained for sample A is illustrated in Figure 4.

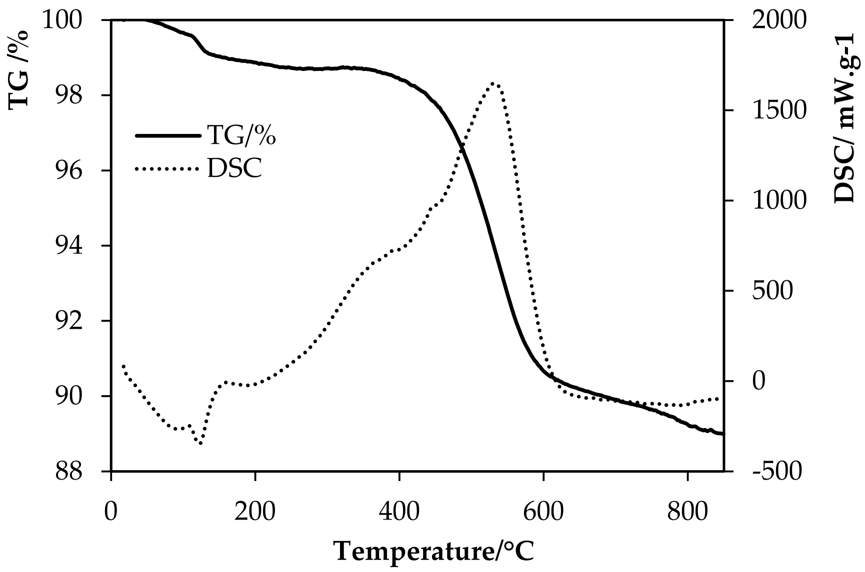


Figure 4. TG/DSC curves of minestone exposed to temperature gradient 30–850 °C under a flow of air (sample A).

Evidently, the most intense oxidation destruction of the sample starts at temperatures of about 500 °C; the steep loss in the sample mass (TG, solid curve) is simultaneously accompanied by marked evolution of the combustion heat (DSC, dashed curve). The temperatures of 500 °C can be considered the level when the smouldering phase turns to uncontrolled flaming combustion of the minestone. One can thus deduce that to spread the fire further to the dump body, the minestone must be heated up to a minimum of approximately 500 °C. Assuming that the mean specific heat capacity of the minestone is 1 kJ (kg·K)$^{-1}$ [16,17], the minimum heat Q_m to increase the minestone (1 kg) temperature to 500 °C (from 30 °C) is Q_m = 470 kJ. From Equation (1), using a net calorific value of 24,500 kJ kg^{-1} for the coal standard, we can read that the minimum combustion heat of 470 kJ can be supplied by minestone containing at least 1.9% of the *EEC*. Thus, the combustible content of ca 2% can be considered the theoretical/minimum limit to evolve sufficient heat to increase the temperature of minestone to 500 °C, at which spreading the fire within a thermally active dump can proceed. Of course, such estimation is simplified with a range of assumptions, like (i) strictly adiabatic conditions of the system; (ii) all particles/inclusions of the combustibles are accessible for oxygen; (iii) minestone is water-free; (iv) there is no restriction in oxygen/air supply to minestone. The following laboratory investigations were then performed to assess a more realistic value of the limiting content for combustibles.

3.2.2. Experimental Investigations

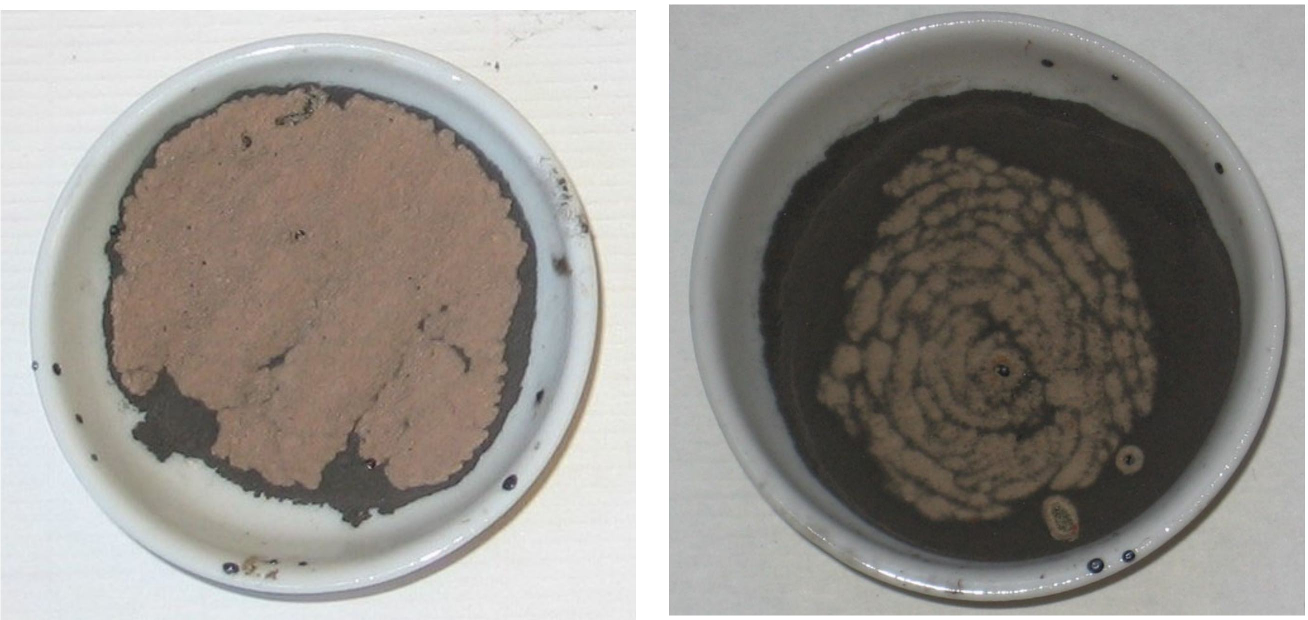
In principle, three basic states of the coal–mineral matter mixtures were observed after the combustion calorimetry test. Mostly, complete burn-out of the sample was evident throughout the whole ceramic plate, with solid encrustation ("cake") of the inorganic residue usually formed, see Figure 5.

Figure 5. Solid inorganic residue (encrustation) of the completely burned-out sample after the combustion calorimetry test.

This was observed for all the samples with combustibles exceeding 10%; for the sample of *EEC* = 30%, the plate was even broken due to oxidation heat. On the other hand, only the cotton fuse was burned in the test with a mixture of 5% combustibles, thus proving that no fire spread through the tested sample (the case is shown in Figure A2, Appendix B). And finally, an intermediate state was observed for the sample containing 7% of combustibles when only a portion of the sample was burned, see Figure 6a.

Based on the observations, the combustibles content of about 7% can be considered limiting with respect to spread/suppression of the fire through the minestone. Measurements then approved the limit of combustibles of other, real samples taken at the Heřmanice coal tailing dump. Thus, samples A and B with *EEC* content of *EEC* 7.4% and 8.5%, respectively (see Table 1), were also confirmed as "limiting", thus expanding the limiting content of combustibles (EEC_m) to a wider interval, EEC_m = 7–9%. Surprisingly, as a minestone of the limiting combustibles content, the sample "R" of the mudstone was also recognised. It was originally taken as a "pure" rock sample (see Section 2.1); however, it proved 6.8% of the *EEC*. The state of sample R after the calorimetric test is illustrated in Figure 6b.

Clearly, the finding on the limit content of combustibles (EEC_m = 7–9%) is valid only for the actual laboratory conditions of the performed experiments. Thus, the scale analysis was made to transfer the finding closer to the real/in situ conditions.



(**a**) (**b**)

Figure 6. Partly burned-out sample after the combustion calorimetry test of the coal–mineral matter mixture sample with 7% of combustibles, (**a**) and the sample "R" of mudstone, (**b**).

3.2.3. Scale Considerations

Table 2 compares experimental conditions under the laboratory calorimetric test and real conditions in a thermally active dump. Parameters for the real conditions were estimated as valid at the Heřmanice coal tailing dump that resulted from the previous investigations [18]. In the final column of Table 2, the expected shift in the limiting content of combustibles EEC_m in relation to the "laboratory" value of 7–9% is denoted.

Table 2. Comparison of parameters for laboratory experiment and in situ conditions affecting the limiting content of combustibles, EEC_m.

Parameter	Laboratory Experiment	In Situ (Heřmanice Dump)	Change in the Limit, EEC_m
Grain size	≤0.2 mm	~0–100 mm	Increase
Accessibility/partial pressure of oxygen	Easy/partial pressure of O_2 = 3000 kPa	Difficult/partial pressure of O_2 = 20 kPa	Increase
Dissipation of heat to the surroundings	Marked: ("pseudoizothermal conditions")	Hindered: ("pseudoadiabatic conditions")	Decrease
Elapsing time of firing	Transient (order of seconds)	Long-term (order of months/years	Decrease
Content of moisture	Minimal (air dried): Moisture < 1%	Moisture ~10% *	Increase (by 1% for 10% of moisture)
Unburned residue of combustibles	Negligible	About 1% *	Increase (by 1%)

*—the value was determined/estimated from previous investigations at the Heřmanice dump [18].

The effect of different moisture content can be evaluated relatively easily. Knowing the evaporation heat of the water to be 2257 kJ kg^{-1} [19], one can estimate that the 10% moisture content increases the limiting content of combustibles EEC_m by 1%. Another 1% increase in EEC_m respects the observations that a minestone of burned-out parts of the Heřmanice coal tailing dump contains roughly 1% of residual/unburned combustibles [18].

Thus, considering the effect of the two parameters, increasing of the value of the limiting content of combustibles EEC_m for practice by ca 2% in comparison with that for laboratory conditions can be estimated.

However, a question arises about how to evaluate the effects of the first four parameters in Table 2, which represent huge (by several orders of magnitude) differences between laboratory and in situ conditions. Due to the practical impossibility of the exact quantification of each parameter individually, we have accepted a simplified solution based on the fact that two of the parameters (specifically, grain size and accessibility/partial pressure of oxygen) lead to the increase in EEC_m value, while the other two parameters (specifically, heat dissipation and elapsing time of firing) lead to the decrease in EEC_m. That is, we ingenuously assume that the adverse/opposed effects of these parameters will lead to the elimination of a possible shift in the value of EEC_m for in situ conditions.

As a result, based on the above considerations, we estimate that the limiting content of combustibles EEC_m for practice will be increased (only) by ca 2% in comparison with that for laboratory conditions, i.e., EEC_m is $10 \pm 1\%$.

In view of the obtained results, it is worth mentioning the calorific value of 7000 kJ kg^{-1} designated earlier by Skarzynska as critical with respect to the inflammability of minestone [9]. Specifically, the obtained content of combustibles $EEC_m = 10 \pm 1\%$ corresponds to the calorific value of the minestone 2205–2695 kJ kg^{-1}. Thus, the proposed limit is about three times lower than that reported earlier by Skarzynska [9].

4. Conclusions

Based on the performed investigations, the following conclusions are regarded as principal:

(i) The content of combustible matter in minestone should properly be evaluated as the energetic equivalent of combustibles, *EEC*, relating oxidation/combustion heat of the combustibles to that of the coal standard.

(ii) As a theoretical limiting content of EEC_m to prevent the spreading of fire in minestone, a value of 2% can be considered.

(iii) For real conditions of thermally active coal waste dump, the value of EEC_m of ca 9–10% was suggested as a boundary between fireproof and fire spreading minestone for conditions of Heřmanice tailing coal dump to be investigated.

Supplementary Materials: The following supporting information on TG/DSC experiments of the investigated samples can be downloaded at: https://www.mdpi.com/article/10.3390/en16135054/s1, Sheet S1: Sample A; Sheet S2: Sample B; Sheet S3: Sample D; Sheet S4: Sample E; Sheet S5: Sample F; Sheet S6: Sample G.

Author Contributions: B.T.: Conceptualization, investigation, writing—original draft. P.G.: Methodology, resources, validation. G.Z.: Investigation, data curation, writing—review and editing. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: The data presented in this study are either available within the article (including Appendix A) or the Supplementary Material Sheet S1.

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Conflicts of Interest: The authors declare no conflict of interest.

Appendix A

Figure A1 displays TG/DSC experimental curves for samples not shown in the main text. (In the tabulated form, the data are available in Supplementary Material Sheet S1).

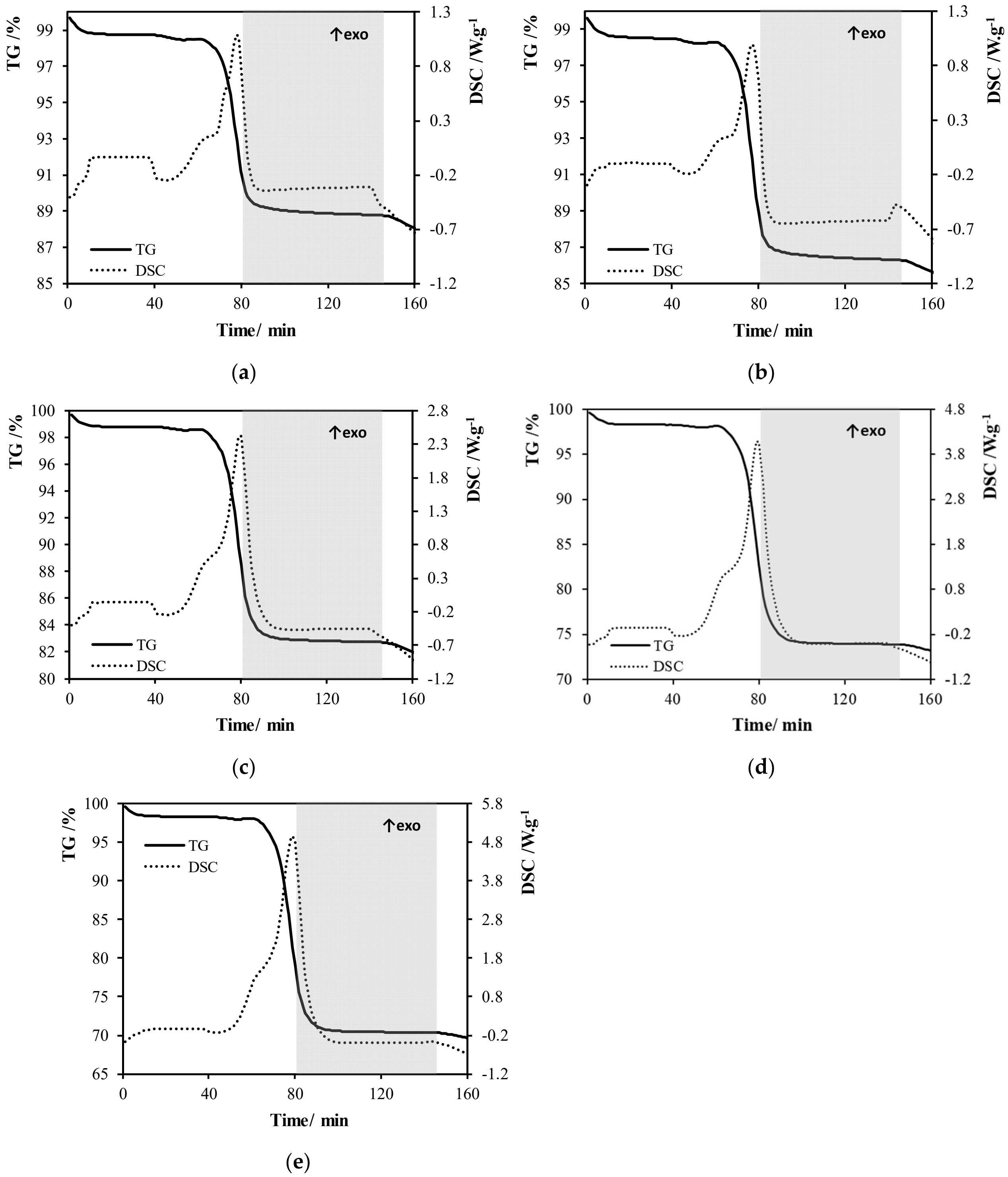

Figure A1. TG/DSC curves of minestone exposed to temperatures 30–550 °C under inert (argon) atmosphere for (**a**) sample A; (**b**) sample B; (**c**) sample D; (**d**) sample F; and (**e**) sample G, where the grey area denotes elapsing time with a constant temperature of 550 °C.

Appendix B

Figure A2 demonstrates the result of the calorimetric test when no fire was spread through the tested sample.

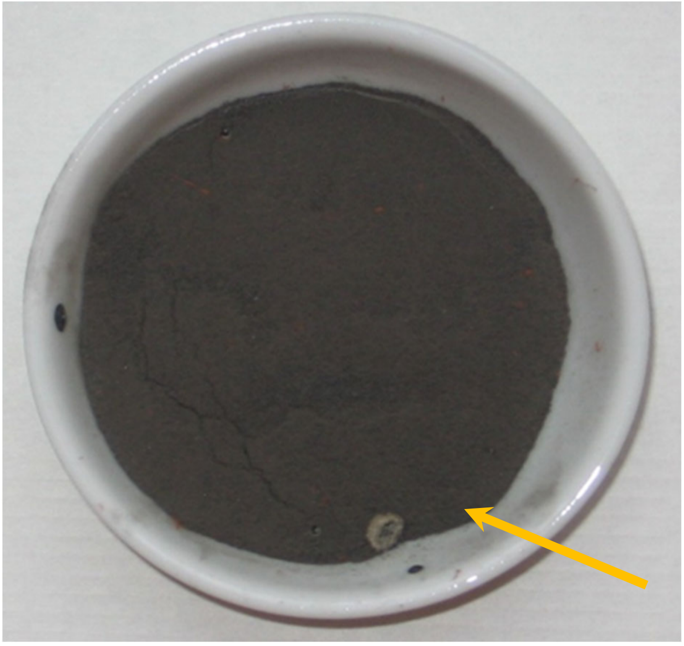

Figure A2. Example of unburned sample after the combustion calorimetric test; the arrow shows initiation point of the fire.

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