

Article

The Impact of Aluminosilicate Additives upon the Chlorine Distribution and Melting Behavior of Poultry Litter Ash

Izabella Maj ¹, Kamil Niesporek ¹, Krzysztof Matus ^{2,*}, Francesco Miccio ³, Mauro Mazzocchi ³
and Paweł Łój ⁴

¹ Department of Power Engineering and Turbomachinery, Faculty of Energy and Environmental Engineering, Silesian University of Technology, 44-100 Gliwice, Poland; izabella.maj@polsl.pl (I.M.); kamil.niesporek@polsl.pl (K.N.)

² Materials Research Laboratory, Faculty of Mechanical Engineering, Silesian University of Technology, 44-100 Gliwice, Poland

³ Institute of Science, Technology and Sustainability for Ceramics, Italian National Research Council, via Granarolo 64, 84018 Faenza, Italy; francesco.miccio@cnr.it (F.M.); mauro.mazzocchi@issmc.cnr.it (M.M.)

⁴ Department of Fundamentals of Machinery Design, Faculty of Mechanical Engineering, Silesian University of Technology, 44-100 Gliwice, Poland; pawel.loj@polsl.pl

* Correspondence: krzysztof.matus@polsl.pl

Abstract: The use of poultry litter (PL) as a sustainable fuel is gaining more attention due to its wide availability and carbon neutrality. However, this type of feedstock is rich in ash and typically contains a high concentration of chlorine (Cl) and alkali elements (Na, K). Therefore, it is likely to cause unwanted issues during combustion and co-combustion, such as chlorine-induced corrosion, ash deposition, and bed agglomeration. In this study, for the first time, the influence of aluminosilicate additives on the above problems of poultry litter was investigated. Three aluminosilicate minerals are under consideration: kaolin, halloysite, and bentonite. Their influence on the chemical composition and melting tendencies of two poultry litter ashes are determined. The investigated ashes, PL1 and PL2, are characterized by different chlorine contents of 6.38% and 0.42%, respectively. The results show that in the case of the chlorine-rich PL1 ash, the additives reduced the chlorine content by up to 45%, resulting in a 3.93% of chlorine in the case of halloysite, 3.48% in the case of kaolin, and 4.25% in the case of bentonite. The additives also positively influenced the shrinkage starting temperature and the deformation temperature of the PL1 ash.

Keywords: biomass; poultry litter; combustion; ash; chlorine corrosion; kaolin; halloysite; bentonite



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1. Introduction

A sustainable energy policy based on low- or no-emission energy sources is crucial for meeting climate commitments. Reducing greenhouse gas emissions from fossil fuel combustion is important to limit the greenhouse effect, and therefore, alternative energy sources such as biomass are gaining interest. This is supported by the fact that in 2022, biomass energy production represented 6% of the world's energy demand, comprising 55% of renewable energy generation [1]. Biomass is a versatile energy carrier, which can undergo various conversion processes, such as combustion, pyrolysis, gasification, or hydrothermal carbonization [2–5].

One of the poorly recognized biomass types is poultry litter (PL). It contains mostly animal manure and bedding material, such as straw or sawdust. The number of poultry farms in Europe is growing due to the high local demand for meat and eggs, together with an increase in the export of these products. According to the data [6], 1.4 billion tonnes of manure from farmed animals were produced annually in EU countries and the UK in the period of 2016–2019.

Poultry litter is traditionally used either directly as fertilizer on agricultural land or composted. As a result, this leads to uncontrolled emissions of methane and ammonia,

lowering local air quality [7]. It also contaminates groundwater with pathogens, antibiotic resistance genes (ARGs), and hormones, as well as other pollutants [8].

Therefore, the issue of animal litter utilization has become vital, and its combustion has been established as one of the most desired disposal paths [9,10]. Consequently, this issue has caught the attention of the European Parliament. According to the regulations of the European Parliament and the Council (EU 2017/1262), the combustion of poultry litter on farm premises is allowed [11]. This fact has already been reflected in a growing number of studies concerning poultry litter combustion, such as the study [12] where the combustion of chicken litter along with a 10% addition of straw or wood is claimed to provide self-sufficiency in heating a poultry farm. In addition to the direct combustion of animal-origin biomass, co-combustion with coal or pyrolysis is also proposed in numerous studies [13–15].

Although biomass combustion technology has been well-known and has accompanied humanity since its beginnings, from a technological point of view, it presents us with a series of challenges and issues. Current power boilers were primarily designed for the combustion of conventional fuels such as coal, which have significantly different physical and chemical properties than biomass. Coal, unlike biomass, typically contains low amounts of volatiles and a high sulfur content [16]. Consequently, the most critical factor for the appearance of boiler corrosion in coal combustion is considered to be the temperature of the sulfuric acid dew point [17,18]. On the other hand, in the case of biomass, which is typically low in sulfur, the main issues with its thermal processing arise from its high content of elements such as potassium, sodium, and chlorine [19,20]. Their presence leads to the formation of chlorides and oxides of alkali metals in the ash, promoting ash agglomeration, and slagging [21–26]. Alkali metal chlorides such as NaCl and KCl, with low melting temperatures, are the main compounds responsible for the high-temperature corrosion of boiler heating elements in biomass combustion [19,20,27,28]. They not only contribute to an increased oxidation rate but also lead to the loss of the protective oxide layer, and the formation of cracks and subsurface corrosion mechanisms [19,27,29]. The corrosion process particularly affects alloys based on the formation of protective chromium-rich oxides. Due to active oxidation, the protective layer degrades [21,30,31]. Additionally, besides corrosion, ash deposition on heating surfaces reduces boiler efficiency. To mitigate corrosion problems in biomass-fired power plants, the working steam temperature is typically kept below 540 °C, resulting in a lower efficiency of electricity generation [19,32].

Tackling slagging and ash agglomeration is crucial for the operation of biomass-fired boilers. Therefore, to predict the risk of ash agglomeration and slagging, authors of numerous studies [33,34] recommend relying on ash fusion temperatures (AFT). In general, the higher these temperatures, the lower the risk of ash slagging.

Although relying on AFT can assist in assessing the risk of ash slagging and agglomeration, it does not constitute a solution for these issues. To limit ash deposition, agglomeration, and corrosion processes during biomass combustion, various fuel additives are applied to influence reactions within the combustion zone. These additives are expected to impact the ash's composition, particularly by reducing its chlorine content, and consequently elevate its melting temperatures. Currently, there is significant research interest in additives based on aluminosilicates, with an emphasis on kaolin [24,28,35–39].

Nevertheless, the impact of aluminosilicates other than kaolin remains poorly investigated. Furthermore, the available literature is solely focused on the applications of additives in the combustion of plant-origin biomass and, according to the authors' knowledge, the influence of aluminosilicate additives on animal litter ash has not been tested yet. Therefore, the presented research covers a knowledge gap and provides novel information, that may increase interest in animal-origin biomass utilization. The work aims to determine the influence of three aluminosilicate additives: kaolin, halloysite, and bentonite on the ash properties of two types of poultry litter collected from European poultry farms. The influence of the equal doses of additives on the ashes' characteristics is determined with laboratory characterization, as a first step before the implementation of the process,

taking into account the ashes' chemical composition, chlorine content, melting tendencies, phase composition as well as microstructural characteristics. The presented research can contribute to the increase in the interest in animal waste thermal conversion, therefore increasing the share of renewable energy in the global energy market and strengthening energy production in rural areas.

2. Materials and Methods

2.1. Poultry Litter

Two types of poultry litter, PL1 and PL2, were subjected to investigation in this study. The samples were collected from poultry farms located in central Europe. The samples were air-dried in laboratory conditions at an ambient temperature and milled in a vibrating mill. The samples were characterized in terms of proximate analysis: moisture content via the thermogravimetric method, ash content according to PN-EN ISO 18122:2015 [40], higher heating value (HHV) and lower heating value (LHV) according to PN-EN ISO 18125:2017 [41], as well as an elemental (ultimate) analysis of carbon (C), hydrogen (H), and nitrogen (N) contents according to PN-EN ISO 16948:2015-07 [42], sulfur (S) and chlorine (Cl) contents according to PN-EN ISO 16994:2016-10 [43].

According to the proximate analysis presented in Table 1, sample PL1 is characterized by an ash content of 15.8% (a.r.), an HHV of 16.93 MJ/kg (d.b.), and an LHV of 15.89 MJ/kg (d.b.). Sample PL2 is characterized by a significantly higher ash content of 48.9% (a.r.), which is reflected in lower values of the HHV and LHV, reaching 11.79 and 10.96 MJ/kg (d.b.), respectively.

Table 1. Proximate analysis of the investigated samples (a.r.—as received, d.b.—dry basis).

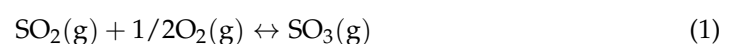
Sample	Moisture	Ash	Higher Heating Value		Lower Heating Value	
	M ^{a.r.}	A ^{d.b.}	HHV ^{d.b.}	HHV ^{a.r.}	LHV ^{d.b.}	LHV ^{a.r.}
	%	%	MJ/kg	MJ/kg	MJ/kg	MJ/kg
PL1	6.8	15.8	16.93	15.78	15.89	14.81
PL2	8.7	48.9	11.79	10.76	10.96	9.79

According to the elemental analysis presented in Table 2, both the PL1 and PL2 samples contain nitrogen at a relatively high level of 1.53% and 2.60%, respectively. They are characterized by various sulfur contents, 0.09% in sample PL1 and 0.36% in sample PL2, while the chlorine contents are similar, being 0.56% in sample PL1 and 0.47% in sample PL2.

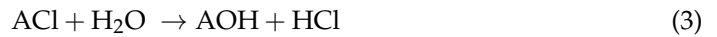
Table 2. Elemental (ultimate) analysis of the investigated samples (dry basis) together with their Cl/S molar ratios.

Sample	C	H	N	S	Cl	Cl/S
	%	%	%	%	%	-
PL1	43.8	4.74	1.53	0.09	0.56	5.64
PL2	30.3	3.85	2.60	0.36	0.47	1.18

The investigated fuels significantly differ in their Cl/S molar ratios, which are calculated as 5.64 for PL1 and 1.18 for PL2. The Cl/S ratio is of great importance due to sulfate formation, a reaction of sulfur with alkali chlorides, resulting in alkali sulfates [44,45]. Sulfur in flue gas mainly exists as SO₂ and to a lesser extent as SO₃ [44]. The equilibrium and reaction rate equations describing this are as follows:



Alkali chlorides have a high ability to react with both SO_3 and SO_2 according to the following reactions (2)–(6) [44,46]:



where A = Na or K.

Therefore, a high sulfur and sulfate content in fuel is expected to reduce the chlorine content in its ash deposits [47,48], thereby limiting the rate of ash deposition and corrosion [47,48].

2.2. Fuel Additives

In this work, three commonly occurring, natural aluminosilicate minerals were selected to serve as fuel additives: kaolin, halloysite, and bentonite. The additives are materials of the clay category whose characteristics and applications were elaborated in detail in [49]. The scanning electron microscope (SEM) pictures of halloysite, kaolin, and bentonite used in this investigation are presented in Figures 1–3.

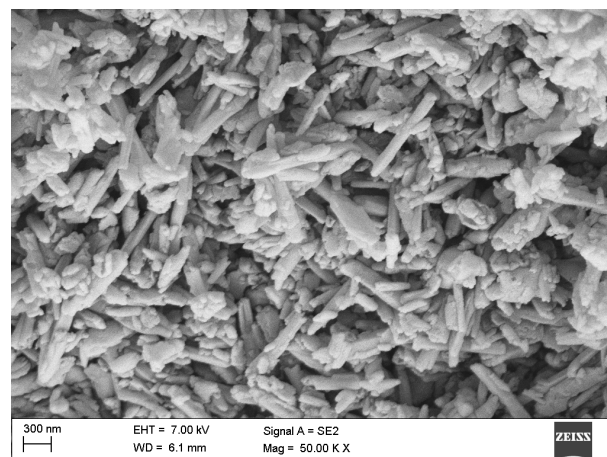


Figure 1. Scanning electron microscope (SEM) image of halloysite [49].

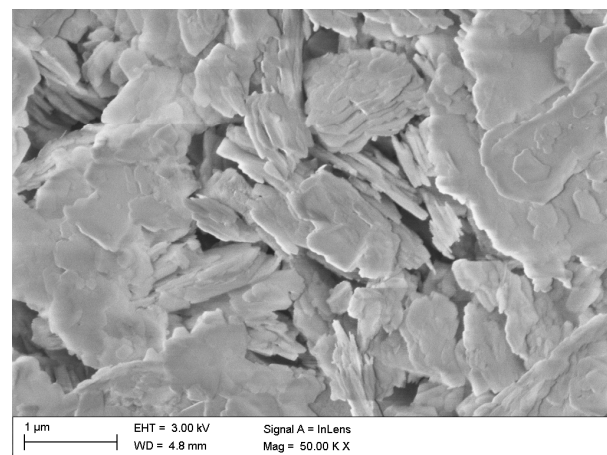


Figure 2. Scanning electron microscope (SEM) image of kaolin [49].

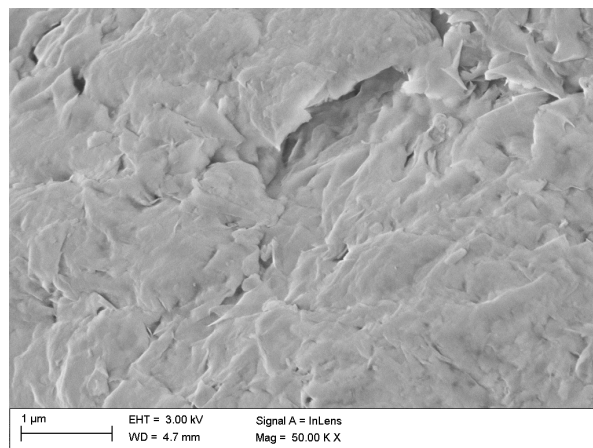
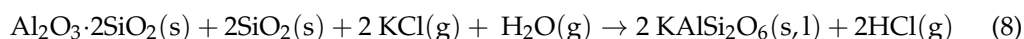
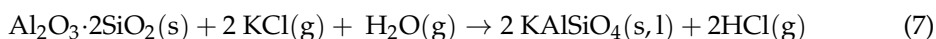


Figure 3. Scanning electron microscope (SEM) image of bentonite [49].

The presented structural analysis of halloysite samples revealed the presence of halloysite nanoplates (HNPs) and halloysite nanotubes (HNTs), while kaolin is a layered silicate mineral that features a predominantly plated structure. Bentonite is a clay consisting mostly of montmorillonite, which, similarly to kaolin, features a plated structure.

Aluminosilicate clays have been considered to be fuel additives due to their favorable characteristics: their easy and safe application, high specific surface area, high porosity, and consequently, high reactivity [50,51]. Studies do not reveal any negative impact of aluminosilicates on the combustion process, such as limitations in their efficiency or generation of pollutants [52,53]. Industrial-application aluminosilicate minerals are chemically stable powders, which results in their convenient application and transportation. Kaolin, halloysite, and bentonite are widely available and do not necessitate costly handling before application. [54].

The application of the aluminosilicate clay powder to the combustion process leads to bonding alkalis in compounds with high melting points, while chlorine is released into flue gas as hydrogen chloride (HCl) [29,55]. According to reactions (7) and (8), aluminosilicates undergo a reaction with potassium chloride (KCl), a compound with a melting point of 770 °C, resulting in the formation of kalsilite (KAlSiO₄) and leucite (KAlSi₂O₆) with melting points of above 1600 °C and 1500 °C, respectively. Aluminosilicates are proven to react with other potassium compounds: potassium sulfate (K₂SO₄) and potassium carbonate (K₂CO₃) according to reactions (9) and (10). In addition to reactions (7)–(10), the bonding of alkalis can be a multi-staged process, starting at a temperature range below the combustion zone, via the adsorption of the alkali metals on the aluminosilicates' surface [56].



The presence of aluminosilicates in the combustion zone leads to the mitigation of unwanted issues connected to the presence of alkalis and chlorine, bed agglomeration in fluidized beds, ash deposition, slagging and fouling on boiler heating surfaces, but also chlorine-induced corrosion by limiting the concentration of chlorine in the ash deposits.

2.3. Ash Preparation and Analyses

The examined poultry litter samples underwent incineration in an air atmosphere in an electric furnace within a consistent temperature zone of 550 °C. The following ash samples were prepared: pure PL1, PL1 with halloysite, PL1 with kaolin, PL1 with bentonite, pure PL2, PL2 with halloysite, PL2 with kaolin, and PL2 with bentonite.

The stoichiometric ratio of the additive can be calculated based on the chlorine content in the fuel, as performed in [35]. With this method, the stoichiometric ratio of the additive (SR) can be calculated according to Equation (11).

$$SR = \frac{M_{\text{Additive}}}{M_{\text{Cl}_2}} = \frac{258.14 \text{ g}}{70.9 \text{ g}} = 3.64 \quad (11)$$

This method overlooks several factors including the sulfation of alkali metals, the impurities present in the additives, the incomplete homogenization of the fuel-additive mixture, and the losses of the additives during combustion. Additionally, potential discrepancies may arise for fuels with higher chlorine contents compared to alkali metals, a scenario uncommon in plant-derived biomass but relevant in animal-derived feedstock.

For PL1, whose chlorine content equals 0.56%, the SR is calculated as 2.04%, and for PL2, whose chlorine content equals 0.47%, the SR is 1.71%. Although recommended stoichiometric doses are approximately 2%, based on the authors' previous studies with coal and biomass, the dosage for each additive was increased to 8% of the fuel mass for several reasons, mainly the elevated ash content in the poultry litter, its heterogeneity, and the presence of impurities in the additive. Such properties of poultry litter may cause unfavorable reaction conditions, resulting in the need for over-stoichiometric additive doses.

According to the research presented in the literature, aluminosilicates are usually applied to fuels in doses of 1–15%. In the research [57], biomass pellets produced with kaolin are investigated, and the stoichiometric amount was boosted by 10% to guarantee the effective integration of kaolin into the pellets. As a result, an additive level spanning from 0.6% to 2.3% was applied. However, the research concluded that kaolin's influence on combustion was negligible, largely due to its low levels of additive incorporation. In another study [58], higher additive ratios, of up to 15%, were investigated during the combustion of agricultural biomass. In this case, a swift formation of K–Al/Fe silicates was observed, with the trend rapidly increasing at an additive ratio of up to 12% and gradually rising between 12% and 15%. The application of kaolin to olive cake and wood in a dose of 5% was investigated in [28] and a rise in the initial deformation temperatures was observed.

In the presented research, the ashes obtained by the incineration of poultry litter with and without additives were subjected to elemental analysis, ash fusion temperatures (AFTs) determination, microstructural characterization, and X-ray diffraction (XRD) phase analysis.

The ashes' chemical composition was determined using inductively coupled Plasma-optical emission spectrometry (ICP-OES).

Ash fusion temperatures were determined under oxidizing conditions through the microscope-photographic method according to standard ISO 21404:2020-08 [59]. This process involved identifying and recording the shrinkage starting temperature (SST), deformation temperature (DT), hemisphere temperature (HT), and flow temperature (FT). The maximum testing temperature was 1500 °C.

The structure and morphology of the powders were examined using a high-resolution scanning electron microscope (HRSEM). The examinations were conducted using a Supra 35 scanning electron microscope from Zeiss (Jena, Germany) with an accelerating voltage of 20 kV and magnifications ranging from 100× to 50,000×, equipped with an X-ray detector with energy dispersion (EDS), model EDX UltraDry from Thermo Scientific™ (Waltham, MA, USA). The powders intended for the study were coated with a layer of gold approximately 15 nm thick to dissipate the electric charge.

X-ray powder diffractometry analyses were performed on fine powders in PMMA sample holders minimizing orientation effects. A Bruker D8 Advance (Bruker Co., Billerica, MA, USA) diffractometer with a high-speed SSD Lynxeye detector was used (Theta-Theta Bragg-Brentano geometry configuration and CuKα₁ radiation), with a 5–80 2-θ range; step: 0.02 deg/s; time: 0.5 s (≈1.5 min equivalent time).

3. Results and Discussion

3.1. Influence of Additives on Chlorine Distribution in Ash

The elemental compositions of the investigated ashes are displayed in Table 3. The chlorine contents have been recalculated taking into account the dilution of ashes with additives (Cl^{rec}).

Table 3. Elemental compositions of the ashes (dry basis).

Sample	Cl	Cl^{rec}	SO_3	K_2O	SiO_2	Fe_2O_3	Al_2O_3	Mn_3O_4	TiO_2	CaO	MgO	P_2O_5	Na_2O	BaO	SrO
PL1	6.38	6.38	1.37	11.2	59.20	2.60	3.51	0.12	0.25	6.53	2.20	5.02	0.68	0.03	0.02
PL1 halloysite	3.42	3.93	1.04	8.40	56.20	6.95	8.57	0.21	1.07	5.45	1.94	4.19	0.68	0.05	0.02
PL1 kaolin	2.97	3.48	0.98	8.53	58.90	2.34	12.30	0.09	0.37	5.41	1.83	4.06	0.58	0.03	0.02
PL1 bentonite	3.74	4.25	1.10	8.55	58.90	2.65	7.00	0.11	0.23	5.63	2.41	4.06	4.55	0.04	0.02
PL2	0.42	0.42	1.30	6.10	65.90	1.90	4.80	0.08	0.26	5.30	1.70	5.20	0.80	0.03	0.01
PL2 halloysite	0.30	0.46	1.20	6.90	60.70	5.20	8.00	0.17	0.87	5.30	1.60	3.90	0.75	0.05	0.02
PL2 kaolin	0.22	0.38	1.10	6.40	66.60	1.70	8.90	0.07	0.31	5.00	1.50	4.20	0.61	0.03	0.01
PL2 bentonite	0.33	0.49	1.00	6.80	65.30	1.90	6.20	0.08	0.21	5.90	1.80	3.40	1.26	0.05	0.02

As presented in Section 2.1, a higher sulfur content in the fuel can contribute to reducing the chlorine content in the ashes. This analogy is illustrated by the analysis of Figure 4. Fuel PL1 with a high Cl/S ratio shows significant chlorine content in the ash—above 6%. Fuel PL2, with a similar chlorine content but a significantly higher sulfur content, has a much lower Cl/S ratio, which results in a much lower chlorine content in the ash, not exceeding 0.5%.

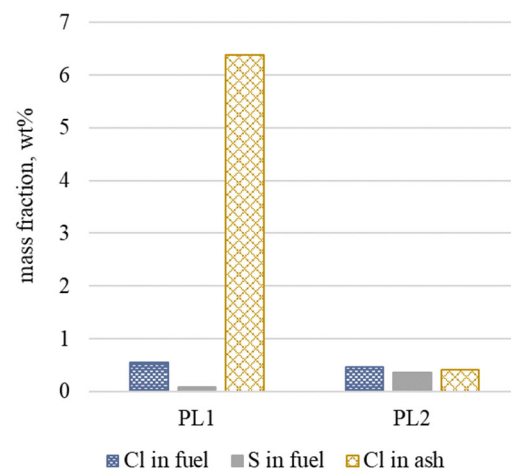


Figure 4. Chlorine and sulfur contents in the fuel samples together with the chlorine content in the ash (dry basis).

The influence of additives on the chlorine content in ashes is presented in Figures 5 and 6. For the PL1 ash, whose initial chlorine concentration in ash is extremely high, it has been visibly reduced as a result of the additives' presence. Chlorine content reductions of 38.46%, 45.51%, and 33.44% are observed in the cases of halloysite, kaolin, and bentonite additions, respectively, being the net of the ash dilution effect due to the additive. The release of chlorine into the gaseous phase took place as a result of the alumina-silication of potassium chlorides. The chlorine is expected to be released in the form of HCl according to Equations (7) and (8).

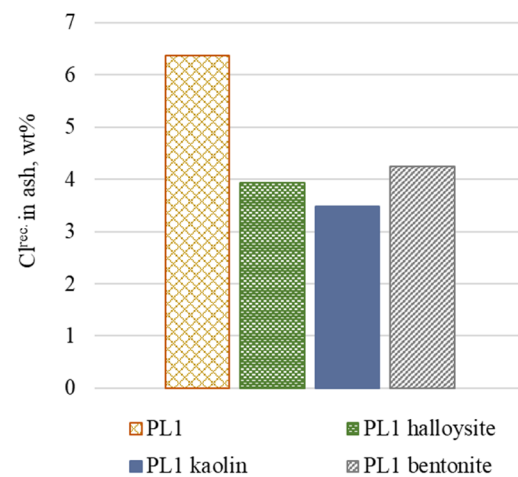


Figure 5. Recalculated chlorine contents in PL1 ashes.

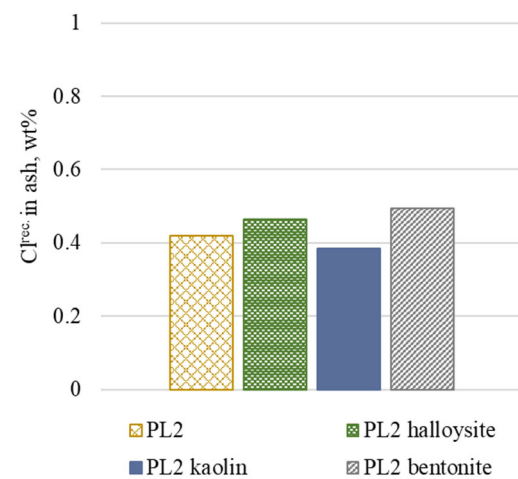


Figure 6. Recalculated chlorine contents in PL2 ashes.

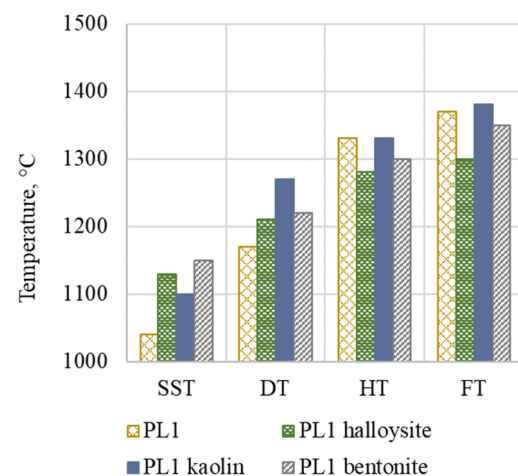
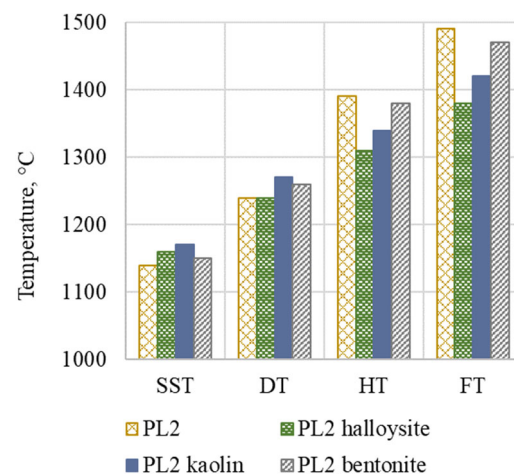
For the PL2 ash, whose initial chlorine concentration is significantly lower, the influence of additives is not clear. In the case of kaolin, a reduction of 8.67% is observed, whereas bentonite and halloysite have no positive effect. Two factors are likely to cause the absence of a clear chlorine reduction. First is the high ash content in PL2 (48.9%), which results in the dilution of the reagents, causing unfavorable conditions for the reactions to occur. Second is the already low chlorine content in the PL2 ash (0.42%), which is a result of the chlorine sulfation reactions taking place during the incineration phase.

3.2. Influence of Additives on Ash Fusion Temperatures

The shrinkage starting temperature (SST), deformation temperature (DT), hemisphere temperature (HT), and flow temperature (FT) of the investigated samples are presented in Table 4 as well as Figures 7 and 8.

Table 4. Ash fusion temperatures of the investigated samples.

Sample	SST	DT	HT	FT
PL1	1040	1170	1330	1370
PL1 halloysite	1130	1210	1280	1300
PL1 kaolin	1100	1270	1330	1380
PL1 bentonite	1150	1220	1300	1350
PL2	1140	1240	1390	1490
PL2 halloysite	1160	1240	1310	1380
PL2 kaolin	1170	1270	1340	1420
PL2 bentonite	1150	1260	1380	1470

**Figure 7.** Ash fusion temperatures of the PL1 ashes.**Figure 8.** Ash fusion temperatures of the PL2 ashes.

For the PL1 ash, whose shrinkage starting temperature and deformation temperature values are lower than those of the PL2 ash, the presence of additives elevates them. However, the performance of additives in the case of the PL2 ash is not clear, but, what needs to be emphasized is that the SST and DT temperatures of PL2 are already high and comparable to those of coal. No positive change in the hemisphere temperatures and flow temperatures of both the PL1 and PL2 ashes can be observed. These tendencies are in line with other research on the influence of aluminosilicate additives on the AFT of various fuels, where, in the case of low-melting biomass ashes, the clear positive influence was observed by Sobieraj et al. [35], but no clear positive influence was determined in the case of the coal ashes with AFT temperatures exceeding 1100 °C tested by Wang et al. [60].

Therefore, it can be concluded that the performance of additives is particularly effective in the conversion of fuels with low melting temperatures.

3.3. Influence of Additives on Ash Morphology

Figure 9 depicts the PL1 ash with additions of halloysite, kaolin, and bentonite. In comparison, the PL2 ashes appear to be more heterogeneous and irregular, with partially idiomorphic crystalline particles, small platelets, and minute granules in clusters (Figure 10a).

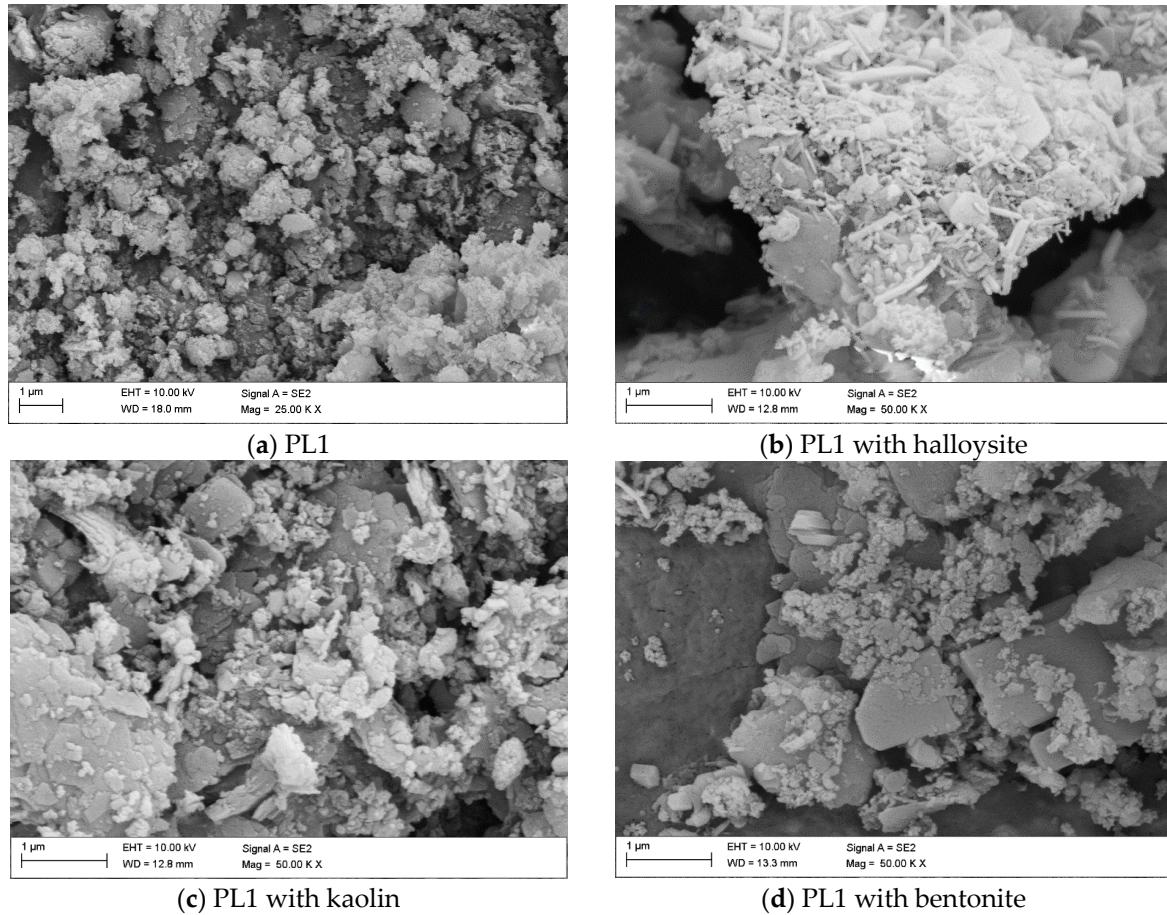


Figure 9. SEM pictures of the PL1 ashes without additives (a), with halloysite (b), with kaolin (c), and with bentonite (d).

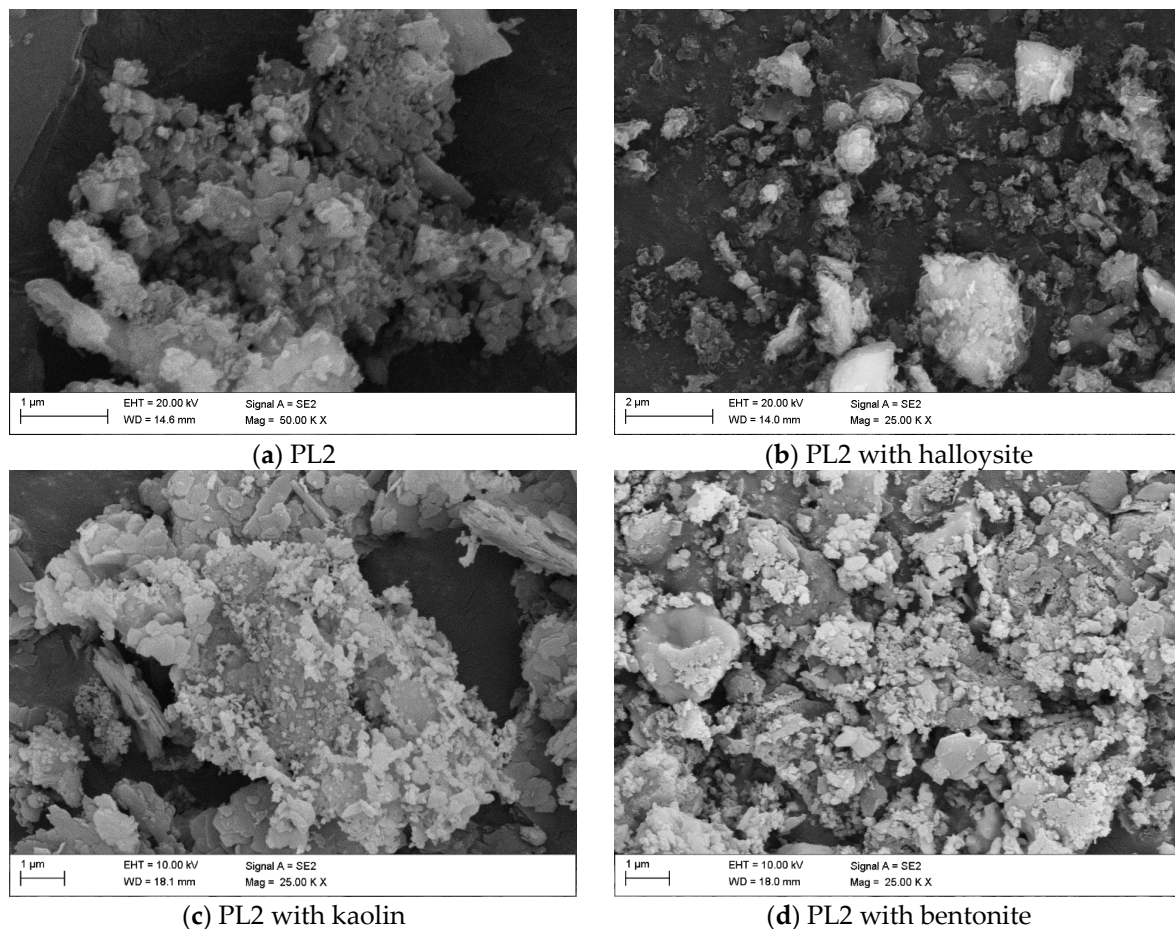


Figure 10. SEM pictures of the PL2 ashes without additives (a), with halloysite (b), with kaolin (c), and with bentonite (d).

In the case of the halloysite addition (Figure 9b), a significant presence of the typical tubular crystals is observed primarily on the surface of the ash agglomerates, where the halloysite has adhered. Also, bentonite seems to allow a certain adhesion of particles and agglomerates onto the platelets of PL1 ash (Figure 9c).

In opposition to this, in Figure 10b, the interaction between PL2 ash and halloysite seems to be scarce probably due to the predominant ash amount in the ash-additive blending.

The electrical status of the clay surfaces interacting with one of the ash particles should also be taken into account: the possibility of the presence (or not) of such weak interactions may be effective in obtaining a homogeneous distribution especially when the added clay is in a smaller amount vs. that of the ash and when particular crystals' morphologies (e.g., for halloysite and its tubular crystalline shape) may be a distinctive characteristic.

Kaolin platelets, kaolin sheets, and even packed sheets with adhered ash particles are present for both PL1 and PL2 (Figures 9c and 10c), leading to a rather homogeneous distribution of the two materials. Kaolin seems to yield better results in terms of reciprocal distribution and homogeneity.

Concerning the PL1 with bentonite particles, mixtures seem to be less effective, showing a clear distinction between the two separate fractions (Figure 9d). A noticeable difference in the better blending of bentonite with the PL2 ash can be noted in Figure 10d, again attributable to the larger ash content of PL2. In this micrograph, the ash particles seem to surround and lay stuck on the bentonite platelets.

The performance of aluminosilicate additives in the ash deposits may be more visible in a higher temperature range, as presented in the work by Hardy et al. [61]. After

the thermal conversion at 1000 °C, the effectiveness of the chlorine removal produced by kaolin, halloysite, and bentonite was significantly elevated compared that to 600 °C. However, the temperature of 950–1000 °C is claimed to be the highest favorable due to the unwanted high-temperature transformation of aluminosilicates [29]. The porosity of aluminosilicate particles decreases at higher temperatures, thereby limiting the pore diffusion of gaseous alkali chlorides into the particles. What is more, in a temperature above 950 °C, meta-kaolinite dissociates into amorphous silica and alumina-silica spinel, which transfer converts into pseudomullite at 1000 °C and both alumina silica spinel and pseudomullite have low potential to react with KCl [62,63].

3.4. Influence of Additives on Phase Composition

The X-ray diffraction (XRD) patterns of the investigated samples are presented in Figures 11 and 12 together with the main detected phases listed in Table 5.

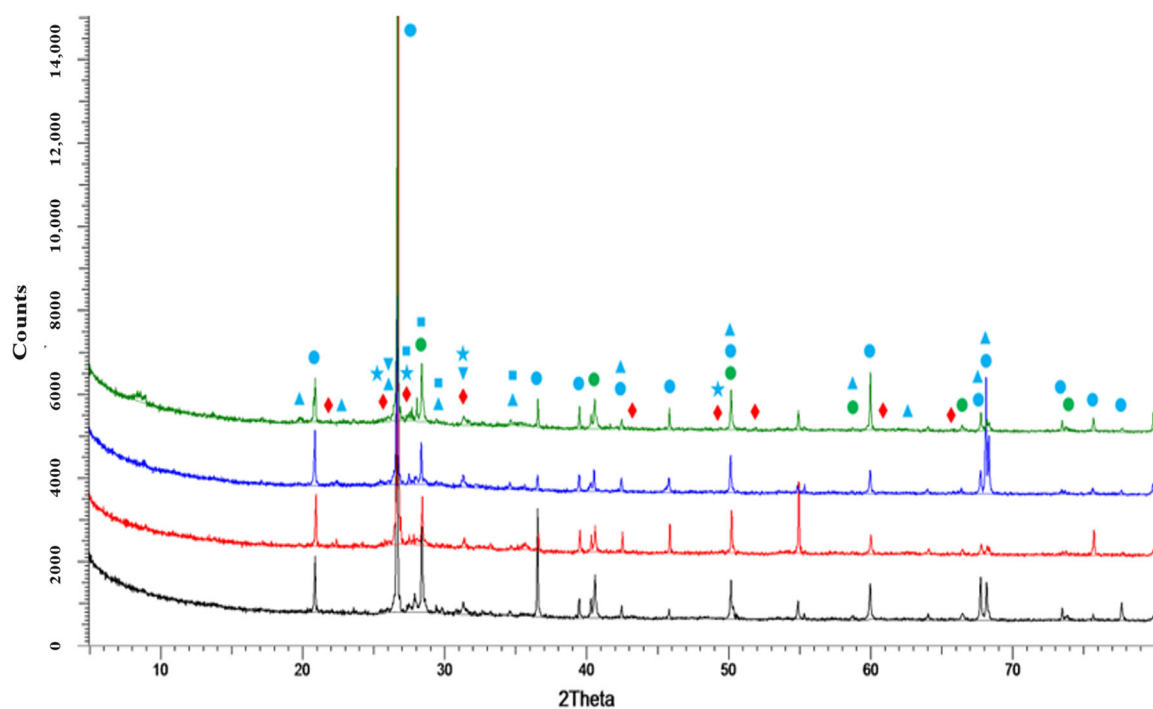


Figure 11. X-ray diffraction patterns of samples PL1 (black), PL1 with halloysite (red), PL1 with kaolin (blue), and PL1 with bentonite (green).

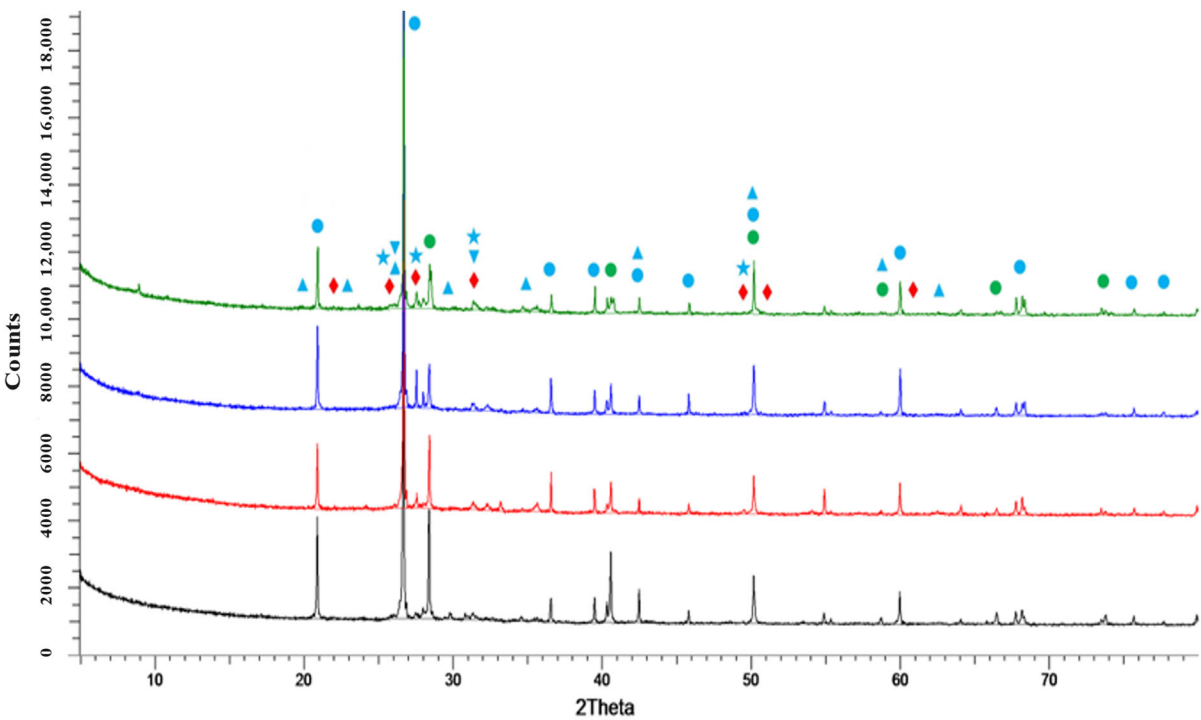


Figure 12. X-ray diffraction patterns of samples PL2 (black), PL2 with halloysite (red), PL2 with kaolin (blue), and PL2 with bentonite (green).

Table 5. The selected main phases detected via X-ray diffraction together with their symbols.

Symbol	Phase
●	SiO ₂ Quartz; Quartz low
●	KCl
◆	Ca-Phosphates (TCMP, Mg-Whitlockite; Cl-Hydroxylapatite)
▲	KAlSiO ₄
▼	KAlSi ₂ O ₆
★	KAlSi ₃ O ₈

The main detected phase in all samples is quartz, which corresponds to the oxide analysis presented in Table 3 as well as what is typical for animal-origin waste [64,65]. Phases containing calcium and aluminum are also detected as feldspar, phosphates, and zeolites, which may indicate the possible use of the investigated ashes in the circular economy, for example, for the synthesis of zeolites [66]. Chlorine is present mainly as potassium chloride (KCl), which is typical for biomass fuels [67]. The presence of KCl is higher in the samples without additives, for both PL1 and PL2 fuels (green dots). In the fuels with additives, the peaks of KAlSiO₄, KAlSi₂O₆, and KAlSi₃O₈ are detected (blue triangles and stars), which is a sign of the reactions between additives and potassium compounds, likely indicating that KCl was dissociated, liberating chlorine in the gas phase.

Also, the presence of some secondary / scarce crystalline phases as metaphosphates and sulfates, and those as mica-like and chain silicates (likely coming from additive phyllosilicates), may indicate the disruption of previous structures (added clay minerals + biomasses) and the rearrangement of new ones.

These latter rearrangements, of course, may be allocating the potassium ions coming from chlorides melting or yielding other crystalline species typical of thermal treatments of biomasses. Those crystalline arrangements are able to coordinate and host the K⁺ ion in

silicate lattices, in between layered structures, in channeled radicals, or by anionic radicals, which could be favored in the range of reached temperatures listed in Table 4. Overall, the XRD analysis would confirm the interaction between ashes and additives.

4. Conclusions

High-temperature corrosion, ash agglomeration, and the slagging of heating surfaces are ash-related problems causing significant concerns in boilers burning or co-burning biomass fuels. When utilizing high-chlorine feedstocks, such as poultry litter, these issues may be particularly severe, and thus, effective methods for their mitigation must be adopted.

In the presented research, for the first time, the influence of kaolin, halloysite, and bentonite on poultry litter ashes has been investigated, taking into account their chlorine distributions and melting tendencies. For chlorine-rich PL1 ash, the chlorine content reductions of 38.46%, 45.51%, and 33.44% are observed as a result of the performance of halloysite, kaolin, and bentonite, respectively. The initial chlorine concentration in the PL1 sample was reduced from 6.38% to 3.93% in the case of halloysite, to 3.48% in the case of kaolin, and to 4.25% in the case of bentonite.

Furthermore, the presence of additives elevates the shrinkage starting temperature and the deformation temperature of PL1 ash, which is expected to make it less prone to agglomeration and slagging. The shrinkage starting temperature and the deformation temperature of PL1 ash were increased from 1040 up to 1150 °C (bentonite) and from 1170 up to 1270 °C (kaolin), respectively.

No clear effect was observed for the PL2 ash, which was rich in ash and low in chlorine, and its ash fusion temperatures were already high. The elevated ash content together with its heterogeneity may cause unfavorable reaction conditions, resulting in the poor performance of additives.

The performance of additives is particularly effective in the conversion of fuels with low melting tendencies and lower ash contents. The presented research covers a knowledge gap and is a preliminary step before the process's implementation, which will be continued with the same approach, taking into account the optimization of the additives' dose as well as various combustion parameters. The first step will be carried out at a laboratory scale, taking into account various doses of the additives and different combustion temperatures. Further investigation on a semi-industrial scale will also include emission measurements with a focus on chlorine's fate and NO_x. Selecting the right dose of fuel additives not only matters for reducing high temperature corrosion effectively but also significantly impacts economic aspects. Previous studies have often overlooked this important aspect. Therefore, in future research, the focus will be on recognizing the influence of individual additives on the economic aspects of the process.

The thermal conversion of poultry litter may be a crucial part of sustainable energy production, especially in rural areas and developing countries, as this type of feedstock is available locally in big quantities. The combustion of poultry litter may provide self-sufficiency in the heating for poultry farms, reducing the consumption of fossil fuels.

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