

INFLUENCE OF IMPURITIES ON THE HIGH-TEMPERATURE BEHAVIOR OF THE LITHIUM-ION BATTERY CATHODE MATERIAL NMC UNDER REDUCING CONDITIONS FOR USE IN THE INDURED REACTOR CONCEPT

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ABSTRACT

In terms of an efficient circular economy in the field of the steadily increasing use of lithium-ion batteries, sustainable recycling methods are of fundamental importance. Therefore, the Chair of Thermal Processing Technology at Montanuniversitaet Leoben has developed the so-called InduRed reactor, a carbo-thermal concept to recover valuable metals from this waste stream. For optimization and further development of this technology, it is essential to have a sound knowledge of the cathode materials' behavior in combination with various impurities in the high-temperature range under reducing conditions. Detailed experiments were carried out in a heating microscope at temperatures up to 1620°C and argon purge. Aluminum from the electrode conductor foils and an excessive proportion of graphite from the anode were identified as the impurities with the most significant negative influence on the process. An optimum melting behavior was found during the tests at an admixture of 10 wt. % C and 1.95 wt. % Al to the cathode material NMC622 ($\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$).

1. INTRODUCTION

The main objective of the international climate policy agreed upon the Paris Climate Conference in 2015 is to limit global warming to below 1.5°C compared to the pre-industrial era. To achieve this goal, industrialized countries must reduce their consumption of fossil fuels and aim for a zero-emissions target by the middle of this century. Concerning the period 2021 to 2030, an overall reduction of 30% on average per country must be achieved in the non-emissions trading scheme (ETS) sectors (Anderl et al., 2019).

A decisive element of the measures to curb climate-damaging emissions is the rapid expansion of renewable energies. A major technical challenge in this respect is the storage of the converted energy. Electricity from renewable energy sources is subject to certain seasonal, regional, and weather-related fluctuations, which is why storage is necessary to implement a sustainable energy economy. In addition to various other technologies, such as Power to Gas and Power to X, battery storage systems will play an increasingly important role (Altmann-Mavaddat et al.; Thielmann et al., 2017).

As part of the European Green Deal, a modernization of the existing battery legislation was proposed at the end of 2020 (European Commission, 2020). With this proposal, the European Union is trying to form a strengthened circular economy to conserve resources and efficiently decouple economic growth from resource dependency. As of July 1, 2024, only batteries for which a CO₂ footprint declaration has been made may be used in the European Union. Furthermore, new targets are set for the content of recycled materials along the entire value chain. To reach these targets, efficient recycling processes are needed that allow most materials to re-enter the material cycle (European Commission, 2020).

Due to the cathode materials' complex structure and chemical composition, the complete recycling process is typically composed of two process steps, one physical and one chemical. The physical process includes pre-treatment steps such as disassembly, crushing, screening, magnetic separation, and thermal pre-treatment. This step significantly reduces the waste's mass and volume in downstream recycling processes, which focus on recovering the valuable metals from the residual stream consisting of active material (or black matter). The black matter

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is composed of anode and cathode material and other components of the LIB structure like electrode conductor foil, which could not be separated completely. The black matter to be treated has the visual appearance of a fine black powder. The following procedure is a chemical process step classified as a pyrometallurgical or a hydrometallurgical process. However, hybrid processes also utilize both pyrometallurgical and hydrometallurgical methods. The hydrometallurgical process typically includes leaching, separation, extraction, and chemical or electrochemical precipitation (Holzer, 2019; Holzer et al., 2021; Huang et al., 2018; Kwon & Sohn, 2020; Swain, 2017). Although this process generally achieves high purities and requires lower energy input, the sensitivity on a fluctuating waste stream composition is a considerable disadvantage compared to pyrometallurgical methods (Holzer et al., 2021).

For industrial-scale applications, processes with pyrometallurgical steps are considered to have higher potential than those with a purely hydrometallurgical approach. This statement is also underlined by the fact that promising pyrometallurgical approaches are already being used in industry (Abdou et al., 2016; Beheshti et al., 2017; Gao & Xu, 2019; Kwon & Sohn, 2020; Li et al., 2016; Sojka et al., 2020; Xiao et al., 2017). However, the considerable need for more optimal process design research is reflected in the disadvantages of pyrometallurgical processes. Since temperatures above 1400°C are necessary for the recovery of the valuable metals, correspondingly high energy input is required. In addition, considerable amounts of waste gas are generated during the process, which must be subjected to downstream waste gas purification. The resulting metal alloy additionally requires a downstream process for use in a closed loop in battery production. However, the most significant disadvantage is that lithium is transferred to the slag phase in currently used methods, from which it is not recovered for functional recycling. (Elwert & Frank, 2020; Huang et al., 2018; Liu et al., 2019; Makuza et al., 2021; Yin & Xing, 2019).

A novel reactor design was developed at the Chair of Thermal Processing Technology at the Montanuniversität Leoben to circumvent this significant issue (Holzer

et al., 2021). The process idea is based on the principle of carbo-thermal reduction of the LIB black matter from a pre-treatment process. For the further development of the process, in-depth fundamental research is required. The fluctuating waste stream and the associated varying chemical composition of the input material pose a particular challenge. Knowledge about the influence of certain impurities on the high-temperature behavior of black matter is of great importance to take optimal advantage of the developed approach. The impurities are residuals that were not separated during the upstream pre treatment procedures. These are mainly non-volatile components such as aluminum, copper, and graphite from the LIB structure. For the investigations presented in this paper, the cathode material NMC622 ($\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$) was chosen. This is one of the currently preferred materials for use in e-mobility (Windisch-Kern, Holzer, Ponak, Hochsteiner, & Raupenstrauch, 2021), with this sector forecast to be and remain a key driver of the technology (Pillot, 2019). A limit value determination of the mentioned interfering substances for optimized use in the InduRed concept is explained in the following. For this purpose, the influences of Al, Cu and C on the cathode material in high-temperature applications under reducing conditions are examined in more detail. Consequently, the behavior in different mixtures is determined.

2. INDURED REACTOR AND ITS REQUIREMENTS

Fundamental knowledge about the desired reactor's properties, benefits, and drawbacks is a prerequisite for comprehending the scope of the presented work. Thus, this part of the paper briefly introduces the InduRed reactor concept, focusing on its potential application for LIBs.

2.1 InduRed reactor scheme

Originally designed and developed to enable superior phosphorus recovery rates from sewage sludge ashes, the InduRed reactor concept, shown in Figure 1, proved itself a promising alternative for several industrial and municipal wastes.

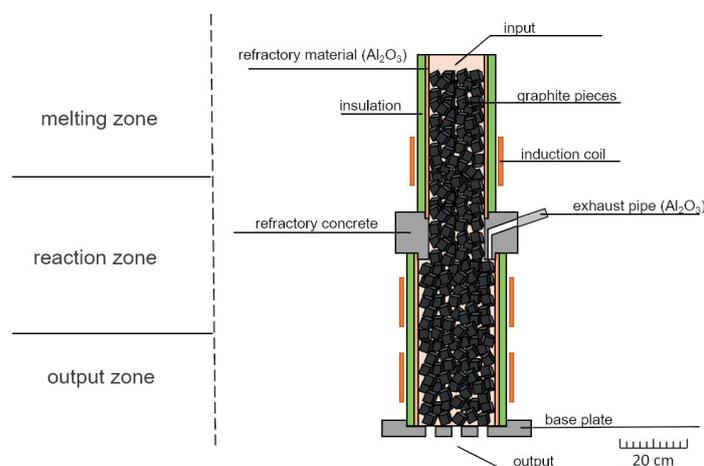


FIGURE 1: Schematic illustration of the continuously charged, fixed carbon bed reactor referred to as InduRed concept (Ponak, 2019).

The InduRed reactor consists of a stack of aluminium oxide rings filled with a fixed bed of graphite pieces and surrounded by three induction coils. An electromagnetic field generated by the induction coils induces a current in the graphite pieces heated to up to 1750°C. It should be noted that the graphite pieces in the reactor serve only as a susceptor material for the inductive heat input, and C powder is added to the feedstock as a reducing agent. The metal oxides containing feed are continuously charged from the top, melt, move downwards, and constantly discharge at the reactor's bottom. Reduction reactions in the reaction zone are particularly promoted due to high CO/CO₂ ratios, a significant reaction surface and a sufficient supply of carbon. The exhaust gas pipe gives the unique opportunity to remove gaseous reaction products directly and thus limit undesired reactions between the gaseous and the liquid phase.

Regarding LIB recycling, the concept should enable the simultaneous recovery of all cathodic metals, including lithium. This is intended to be achieved by separating Li via the gas stream instead of being undesired slagged as in conventional pyrometallurgical procedures.

So far, experiments with pure cathode material from battery production with the addition of carbon in a lab-scale model of the InduRed reactor have already revealed promising results, in which up to 95% of the initial Li was removed from the residual material. In addition, initial results from the investigation of the phosphorus-containing cathode material LFP (LiFePO₄) have also shown that over 64% phosphorus could be removed (Holzer et al., 2021; Windisch-Kern, Holzer, Ponak, Hochsteiner, & Raupenstrauch, 2021; Windisch-Kern, Holzer, Ponak, & Raupenstrauch, 2021; Windisch-Kern, Holzer, Wiszniewski, & Raupenstrauch, 2021).

3. DETERMINATION OF MATERIAL BEHAVIOR IN HIGH-TEMPERATURE APPLICATIONS

For the further development of the InduRed concept towards the waste stream from LIBs, specific knowledge of the material used is the basis for efficient upscaling. Although the InduRed reactor concept presented is capable of withstanding temperatures of up to 1750°C, the target temperature set for using materials from LIBs is about 1550°C. This temperature was chosen because the melting temperatures of the reduced metals contained in the reactor are below 1500°C and a safety margin had to be included due to possible local temperature differences. Thus, in-depth investigations of the material to be utilized in the high-temperature area of 1550° are necessary. The material used as well as the methods are explained in detail below.

3.1 Materials and Methods

To extend the investigations of the high-temperature behavior of the cathode material NMC622 under reducing conditions published by Windisch-Kern et al. (2021) by thermogravimetric analysis and differential scanning calorimetry, experiments in the heating microscope were carried out within the scope of this work. For this purpose, the

TABLE 1: Heating program in the heating microscope to perform the tests with NMC622 and addition of different additives.

Temperature range	Heating rate
Start - 1350°C	80°C/min
1350 - 1450°C	50°C/min
1450 - 1700°C	10°C/min
1700°C	5 min holding time

Hesse Instruments EM 201 with an HR18-1750/30 furnace was deployed. The cathode materials used in the tests are manufactured by and purchased from Gelon Energy Corporation in Linyi, China. These experiments aimed to visualize the changes of the cross-sectional area of the samples over temperature, thus allowing conclusions about the melting ability, which is a requirement of the InduRed concept. To extend these findings towards expected waste stream compositions, additional investigations must be carried out in which possible impurities are added.

For this purpose, different extents of aluminum, copper and carbon were added to the cathode material NMC622. The sample was then pressed in a standardized cylindrical form with an approximate mass of 0.1 g and placed on an Al₂O₃ analysis plate. It should be mentioned that carbon or graphite is used as a reducing agent for the reduction reaction.

For safety reasons, the experiments were not carried out under a CO atmosphere, which would better fit to the actual conditions provided by the InduRed concept. However, since it is primarily essential to prevent oxidation reactions, purging with 2.5 l/min argon was applied instead. Accordingly, the C demand was calculated considering a conversion to CO₂ instead of CO.

Finally, the sample was heated in the heating microscope to an oven temperature of 1700°C, corresponding to approximately 1620°C sample temperature.

The heating program, which can be taken from Table 1, corresponds to the maximum possible rate of the heating microscope used, approximating the range of application in the InduRed concept.

3.2 Experimental approach

The experiments were carried out in two phases to determine the influence of the substances Al, Cu, and C on the melting ability. Firstly, the cathode material was mixed with the elements mentioned above and examined under a heating microscope to assess the behavior. This allowed an estimation of the most significant negative influencing factors. To gain quick information about the successful reduction process without further analysis in the laboratory, the magnetic behavior of all samples was subsequently examined using a neodymium magnet. It should be noted that the stoichiometric carbon demand for complete reduction of NMC622 in inert atmosphere and assuming a conversion to CO₂ is 11 wt. %, rounded. This ratio was taken as the baseline for this series of experiments.

The next phase aimed on determining the limits of elements interfering the InduRed concept requirements of a melting phase. For this purpose, a total of 26 tests were

carried out with different mixing ratios, as shown by the green dots in Figure 2. A specified mass fraction of Al and/or C calculated on the total mass was added to the resulting mass fraction of NMC622 and examined under the heating microscope (Baldauf, 2022).

To be able to make a statement on the change in the cross-sectional area of samples or mixtures other than those investigated and to optimize the number of experiments, an interpolation network was designed, which is shown as a black dotted grid in Figure 2 (Baldauf, 2022).

To evaluate the resulting data quantity of approx. 1500 data points per test accordingly reliable, additional effort is required. Oscillating data areas, which can occur due to optical measurement errors, are corrected by smoothing over a polynomial. Oscillation can also be caused by incorrect detection by the heating microscope due to focusing problems over a more comprehensive temperature range than that. The accrued data gap can be corrected by comparing the last measured values with the stored images and the following linear correlation of the corrected values. Because of the reaction kinetics as well as the adjustment of the temperature ramp of the heating microscope, a specific temperature can address several data points. However, data points may not be available for every temperature, further data processing is necessary. This problem may be overcome by the processing and output of the arithmetic mean of the data of the same temperature values or by a logical continuation of the temperature (Baldauf, 2022).

4. RESULTS AND DISCUSSION

As explained in point 3.2 Experimental approach, the experimental procedure was divided into two parts. A pre-

liminary series of tests was conducted under a heating microscope to determine the basic influence of different impurities. Based on this, a limit value determination of impurities for further pyrometallurgical recovery of valuable metals in the InduRed concept was carried out.

4.1 Basic influence of different impurities

The representation of the cross-sectional area in Figure 3 shows the most important tests in this series. The terminology in the legend, as NMC622_C11_Al10, specifies 11 wt. % C and 10 wt. % Al being added to the resulting proportion of NMC622.

Significant findings can be deduced from Figure 3 comparing the change in cross-sectional area over temperature. Thus, in comparison with NMC622 without addition of C, Al or Cu to the test with the admixture of the stoichiometrically necessary C for complete reduction (NMC622_C11), a significant difference can be seen in the cross-sectional area reduction and in the final area. In addition, the examination of the magnetic behavior showed that only those samples with the addition of C are magnetic. The comparison between NMC622_C11 and those with the addition of Al and Cu is particularly interesting. It can be seen that the end surface of NMC622_C11_Cu10 intersects with the sample without Cu addition. Thus, it can be concluded that the addition of Cu has less a negative influence on the melting behavior since the final surface already develops at lower temperatures, in this case, approx. 1250°C instead of 1450°C. The situation is different for the addition of Al. The essential reduction in the surface area takes place earlier. Still, the final surface is considerably

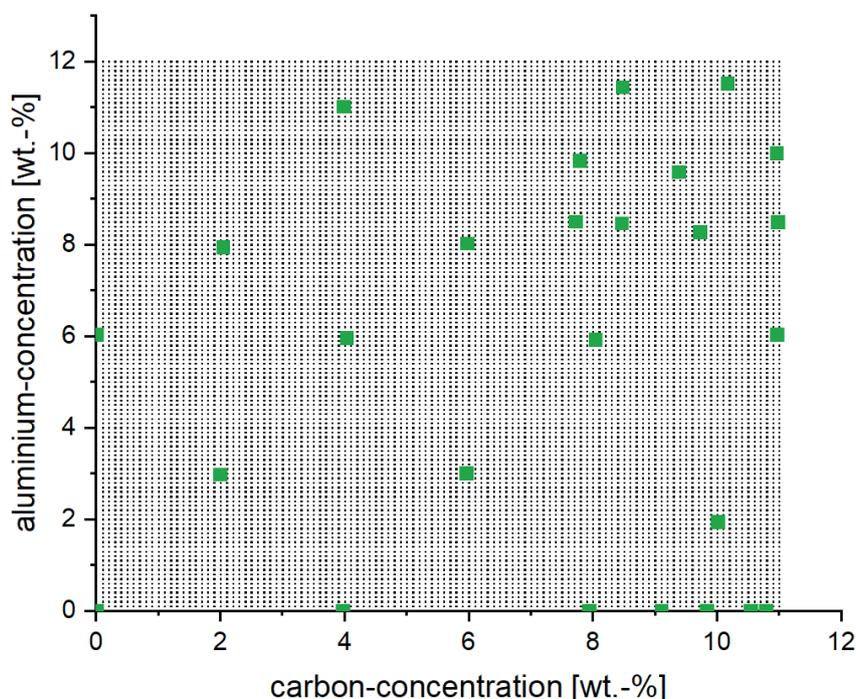


FIGURE 2: Measured points (green points) in the heating microscope depending on the corresponding proportions of aluminum and carbon to NMC622 and the interpolation network (black dotted grid) (Baldauf, 2022).

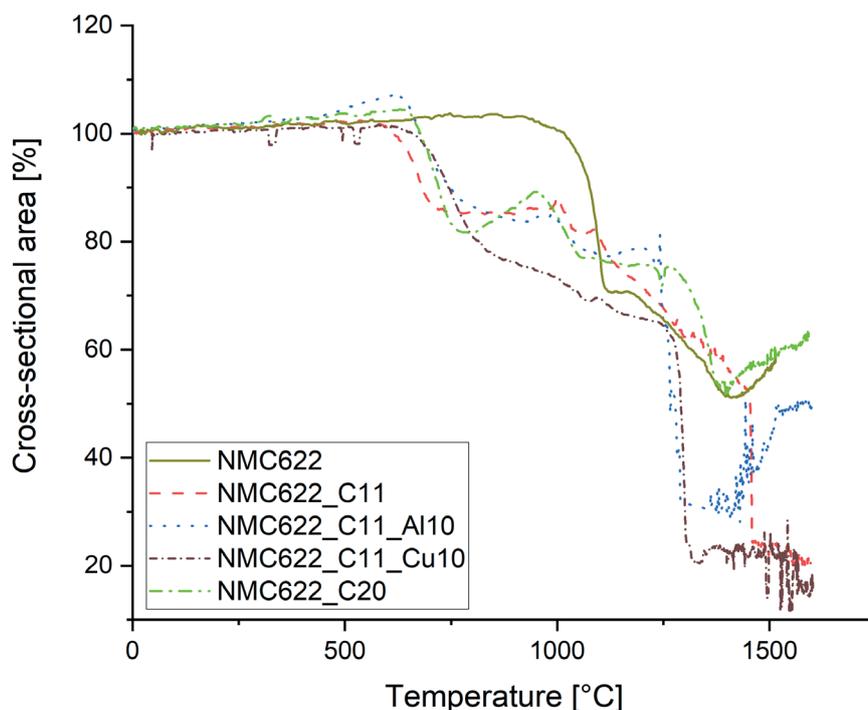


FIGURE 3: Comparison of the change in cross-sectional area in the heating microscope as a function of temperature of NMC622 without and with the addition of Al, Cu and/or C.

higher than that without additives. To gain results on the behavior with the addition of C above the stoichiometrically necessary amount, investigations were carried out with a C content of 20 wt. %. The cross-sectional area's behaviour is considerably worse compared to 10 wt. %. For further detailed studies, it can be deduced that the proportion of Al and C in the mixture has the most significant influence on the melting ability, suggesting intensification of research in this field.

4.2 Limit value determination of interfering substances

Since the rigid two-dimensional view of the cross-sectional area versus temperature for a system of Al/C/NMC622 entails a considerable loss of information, and since it is also possible to represent the high-temperature behavior for other variants of the composition without conducting experiments using the generated data, the vectorial view in three-dimensional space was chosen as the representation variant.

The mesh shown in Figure 4 represents the change in the cross-sectional area of NMC622 in combination with Al and/or C. It can be seen that the area moves towards a composition of 10 wt. % C and 1.95 wt. % Al to an absolute area minimum of 13.59%. To apply this finding to the InduRed reactor, a conversion is necessary, assuming a reaction to mainly form CO instead of CO₂. This results in a C content of 18.20 wt. % to achieve the absolute area minimum.

By comparing the images of the heating microscope and the photos of the sample after the test, it was found

that there is a transition range from a slightly melting fraction to a minimal melting fraction at ranges between 36.6% and 53.3%. Consequently, from the 53.3% mentioned, no more continuous melt was formed. In order to always be able to generate a molten phase in the process, mix ratios should be selected that are in the range of less than 36.6% (Baldauf, 2022).

Resulting from this fact, the data were plotted in a height stratification in a 2-D plot, as shown in Figure 5. A diagonal band from the upper left to the lower right is visible, in which the desired area of less than 36.6% is partially included.

It can be seen that while higher proportions of Al are in principle possible in smaller areas, this is accompanied by lower proportions of C. One possible reason for this is that Al has a higher affinity for oxygen than C, as can be seen in the Richardson-Ellingham diagram (Biswas, 1981). This means that Al acts as a reducing agent (Makuza et al., 2021), which implies that an excess of C inhibits melting. However, it is additionally evident from the figure that the higher melting ability is in the range of lower Al values and C contents towards the stoichiometrically calculated value of 11 wt. % for a complete reduction of the oxides contained in NMC622.

As can be seen in Figure 5, extrapolations were used in the range greater than 6 wt. % Al and 0 wt. % C to 11 wt. % Al and 4 wt. % C. Experiments in this area were not carried out in the present series of tests for safety reasons. This is because of the risk of an aluminothermic reaction observed in parallel trials.

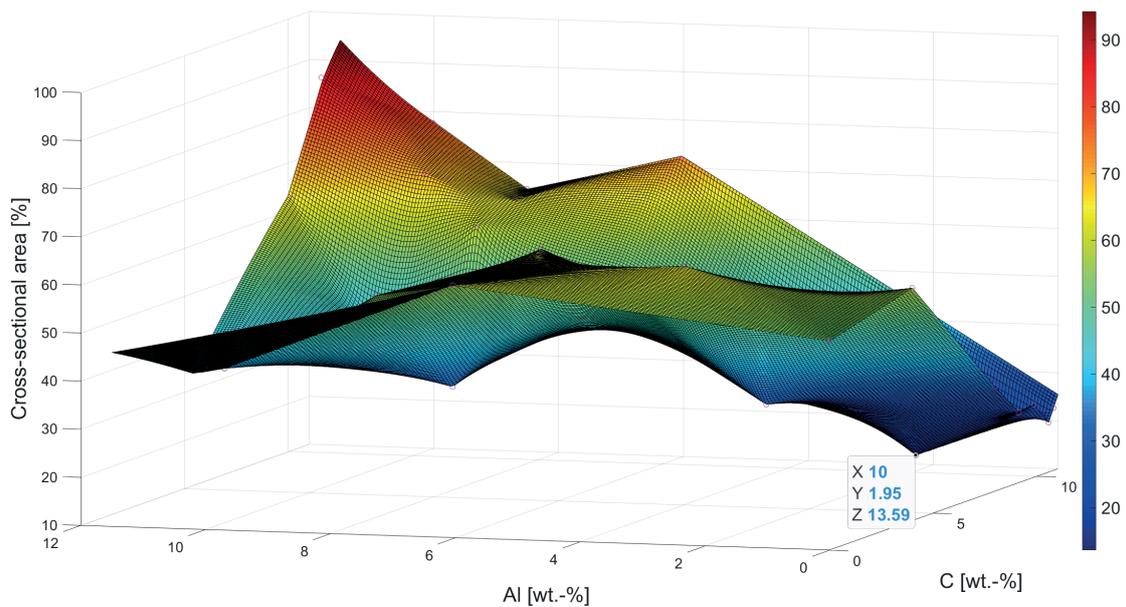


FIGURE 4: 3-D mesh plot of the measured points (blend NMC622 with/without Al or C) in the heating microscope as a function of the cross-sectional area acquired at 1550°C.

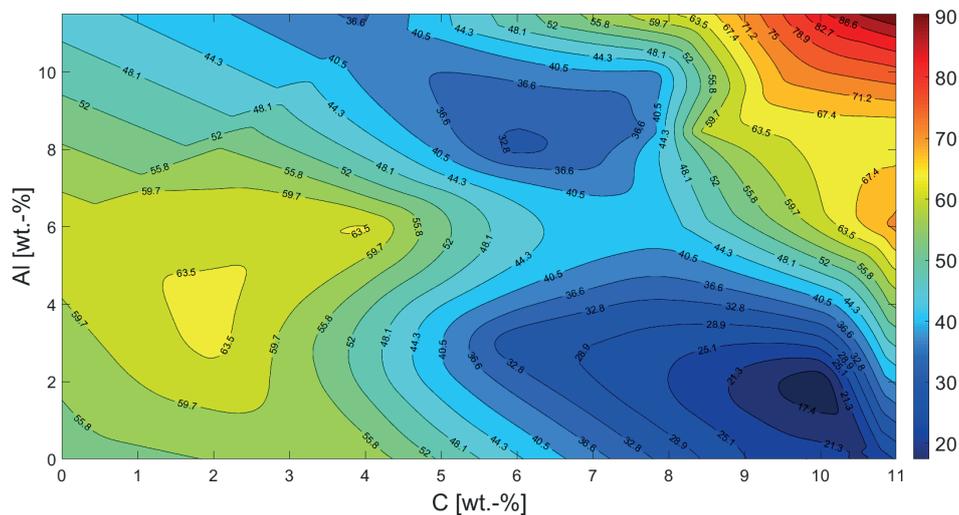


FIGURE 5: Schematic representation of the change in cross-sectional area of the cathode material NMC622 when combined with varying C and Al additions ratios over a height stratification at 1550°C.

5. CONCLUSIONS

In the context of this publication, the compositional requirements of black matter from lithium-ion batteries (LIB) for a pyrometallurgical recycling approach were investigated.

In this respect, it was determined in a heating microscope that the main factors negatively influencing the required melting ability are the elements Al and C. Cu even positively affects the melting temperature in the experimental setup.

Subsequently, the focus was on Al and C. Thus, the change in the cross-sectional area (CSA) at 1550°C with a varying NMC622-Al-C system was investigated. It was found that mixtures with a final cross-sectional area (CSA) of less than 36.6% should be aimed for. Finally, a composi-

tion of 10 wt. % C, 1.95 wt. % Al and the resulting amount of NMC622 with a cross-sectional area of 13.59% was found to be the optimum blend. Considering the reaction sequences in the InduRed reactor, a C content of 18.20 wt. % would be necessary for this respect. In addition, it was recognized that attention must also be paid to the Al/C ratio. Due to their property in terms of oxygen affinity, care must be taken not to result in excess of C, which negatively affects the melting ability. However, even a bunch of Al can cause significant safety and process engineering difficulties with respect to a possible aluminothermic reaction and its strongly exothermic behavior. For this reason, according to initial findings, Al contents of less than 6 wt. % should generally be aimed for, which must be investigated in more detail in further trials.

As an outlook for future research activities, the determination of the properties of all commercially used cathode materials such as NMC, LCO, NCA, and LFP with additional Cu from the electrode conductor foils can be mentioned here. From this knowledge, pyrometallurgical processes can consequently be better adapted to the expected waste stream. An essential point in pyrometallurgy is also the optimization of the resulting products, such as the value-added metal alloy. Attention must be paid to this in the adaptation of input flows, process design, and post-treatment processes development, where in-depth knowledge of high-temperature behavior can significantly contribute.

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