SisAl Pilot

SisAl Pilot Project
Innovative pilot for Silicon production with low environmental impact using secondary Aluminium and silicon raw materials

Enjoy reading the SisAl Pilot newsletter!

Second year project results

Foreword by Coordinator

SisAl Pilot is approaching the end of the second year and thereby also the mid-term of the project. Looking back at the time that has passed many promising results have been produced at different scales by the project partners both on the pyro- and hydrometallurgical side. The mass flow models, LCA models and business cases have been improved, which together with the experimental and modelling results have given us more confidence in the implementation and commercialisation of the SisAl pilot process. This year has also given us many positive memories from meeting partners at different locations in Europe and also at Mintek in South Africa. We hope that you will enjoy reading each partner's summery of their achievements during this year and last but not least we would like to thank all our partners for very fruitful and successful collaboration that we hope and believe will continue during the third and fourth year of the project.

Your sincerely,

Prof. Gabriella Tranell and Dr Maria Wallin

Main updates from the partners

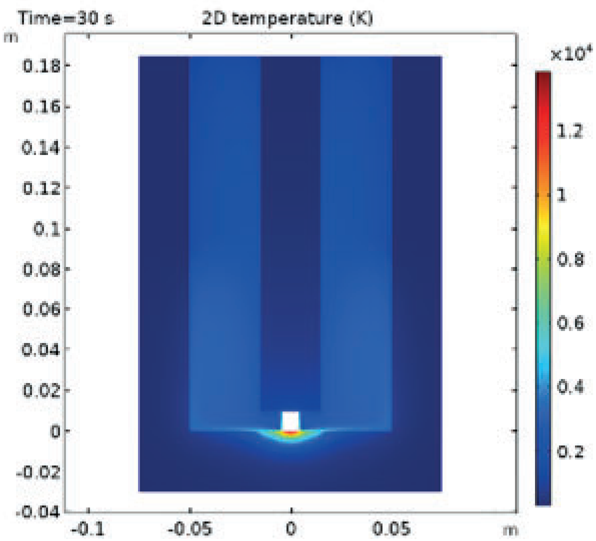
NTNU's focus during the second year of the project has been on performing small scale experiments with the purpose of providing inputs to the pilot trials at Elkem, FRey and long term to RWTH and Mintek. These results have been provided to the pilot sites through discussions and presentations physically and online. The coordinator team has been at Elkem's pilot facilities when they conducted some of the pilot trials and contributed with input to the pilot team. They have also visited RWTHs, FReys and MINTEKs pilot facilities and looked at their pilot set-ups and discussed the upcoming pilot trials.

The LCA team has in collaboration with HZDR finalised the first case study of the SisAl Pilot process which soon will be submitted as a scientific article to Resources, Conservation & Recycling.

Two master students Elisa Pastor Vallés and Gjermund Lie Solbakk have defended their MSc thesis titled "Life Cycle Assessment of silicon metal by aluminothermic reduction: an industrial symbiosis approach" and "The Effect of Varying CaO/SiO2 Ratios and Reductant Addition in Silicon Production by Aluminothermic Reduction of Silica Based Slags", respectively. Elisa is today working as a PhD candidate in SisAl Pilot's LCA team at NTNU and Gjermund is working at Alcoa, which is an aluminium smelter in Norway. Three MSc students are currently working on different topics within SisAl Pilot and they will all deliver their thesis by summer 2022.

NTNU has presented project results at REWAS (at TMS), Infacon XVI, Life Cycle Management Conference (LCM 2021) and will present results at upcoming Silicon XVI conference. Articles in two Norwegian newspapers based on interviews with the project coordinators and the Elkem team have been published.

During the second year of the SisAl project, SIMTEC has worked on improving the modelling of the electric arc. Whereas during the first year, electric arc was considered as an ohmic conductor, a finite-element model based on a Channel Arc Model developed by G. Sævarsdottir, has been developed to consider thermal dissipation due to convection, electron flow and radiation; this model also enables to predict the arc temperature. This model has been implemented to study the first step of the process where only the slag is melted. As the aluminothermic reduction has been decided to be carried out outside the electric arc furnace, in a ladle furnace, SIMTEC has modelled the heat transfer occurring in a ladle furnace. In the developed model, both the power brought by the reaction and the power lost toward the outside are considered. This model enables to predict a temperature distribution inside the system and may help to design the ladle furnace by avoiding early freezing of the slag-metal mix or to avoid too high temperature to be reached.



Temperature field from channel arc model inspired heat transfer model.

MYTILINEOS, as Work-package 3 leader, has coordinated and supervised the slag leaching optimisation experiments at lab scale. It has been shown that the slag produced by Elkem in WP2 is sufficient for both alkaline and acidic leaching. MYTILINEOS has received enough slag for both pilot camping's that will be conducted in the project. All modifications of the alkaline pilot plant have been completed and MYTILINEOS has also design and ordered material to the acidic leaching pilot plant. In 2022, MYTILINEOS will run the alkaline pilot trial with SisAl slag where the goal is to achieve the production of alumina bearing precursor solutions out of which commercial aluminium oxide product can be precipitated.

In the first year, the focus was on finding a suitable process control for the SisAl process, and in the second year, further research was carried out on this subject. The focus was on investigating the influence of different holding times, different raw materials and the properties of the reducing agent on the outcome of the trials. In addition, various preliminary tests were carried out for the planned pilot trials. Although the process could also be carried out in a single-stage process in an electric arc furnace, the tests have shown that a two-stage process consisting of slag production in an electric arc furnace and the reaction of the liquid reactants in a heated ladle is recommended. Due to supply shortages and better feasibility, the pilot trials planned at IME **RWTH** Aachen University will therefore be moved to Elkem's research facilities in Kristiansand and carried out in Q2 2022. These trials will serve as direct pre-trials for the planned campaign at Mintek in South Africa. Thus, the process can already be tested on a smaller scale and adjusted if necessary to guarantee a successful execution of the following trials.

BNW completed several deliverables from WP4 Exploitation and WP7 Management:

D4.1 Prefeasibility study of SisAl Pilot business cases, month 12

D4.2 Key Exploitable Results, month 12

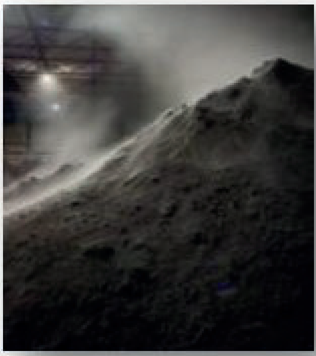
D4.9 SisAl Pilot exploitation roadmap, 2nd version, month 18

D4.4 Innovation database, 1st version, month 18.

D7.9 Innovation management report version 1, month 18

Befesa is responsible for characterizing, selecting and preparing Al based raw materials (WP1) and Befesa secondary Alumina (BSA) (WP3) for the SisAl process.

In the second year, Befesa is working on Pilot 2 (Al-Si alloys). Pilot 2 take place at the high temperature foundry facilities of Frey en Vilagarcia de Arousa, Spain, which include the rotary and induction furnace technology. Al based raw materials (Aluminium concentrates, incinerator bottom ash, aluminum scrap, aluminium crankcase, Zicral shavings) have been provided by Befesa, in order to produce a hyper-eutectic Al-Si alloy. Particle size, chemical analysis before melting, metal content and chemical analysis after melting have been analysed in the Al based raw materials.



Also, Befesa has provided three different types of BSA (Befesa Secondary Alumina) for WP3 (mixed with slag produced in WP2 campaigns which must have a proper mineralogical composition). All these by-products are perfectly characterised.

ERIMSA is currently supplying one of the raw materials (quartz) to the users for the project, as mostly also done last year. The work performed proved the chemical characterization of available different types of quartz and possible sources are by-products presently sold as aggregates for concrete industry or metallurgical quartz for ferroalloys industries.

In SisAl Pilot, ERIMSA is included in the WP 1 In- and output material properties, mixes and analyses with the main focus to select, characterize and prepare quartz from ERIMSA`s mine suitable for the SisAl process. ERIMSA is the main provider of quartz to business case 5. It additionally provided lime to the Spanish company Fundiciones Rey from a Portuguese supplier.

Alkaline leaching and Precipitation

Alkaline leaching of calcium aluminate slags from WP2 is at the final stages of optimization for the pilot work! A calcium aluminate slag produced by aluminothermic reduction in RWTH was used in Na_2CO_3 leaching experiments with the goal of defining optimum leaching conditions for the pilot. For the experimental work, it was the first time that a critical analysis of the existing Pedersen Process patents was conducted and the most suitable Na_2CO_3 concentration for the leaching process was decided. The leaching tests were performed in various temperatures (40-95°C) and s/l ratios (8-12%) and over 80% Al extraction rates were achieved. This research work is described in detail in a scientific paper titled “Critical examination of the leaching operations in the industrial Pedersen Process and application of findings in the leaching of slags produced by a novel aluminothermic reduction process” which was submitted for publishing. Finally, the optimized leaching solution will be further tested by carbonation to precipitate alumina hydrates.

Alkaline aluminate solution produced by the leaching of calcium aluminate slags

Aluminate solutions appear transparent and stable

Laboratory filtration of the residue proceeds without issues

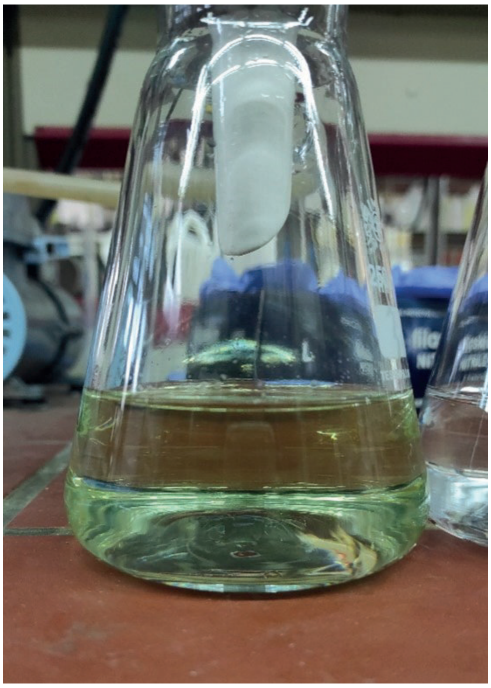
Drying of the residue results in a powdered material. Larger coagulated particles easily break down with force applied by hand.

Acid leaching

Laboratory Acid leaching of calcium aluminate slags from WP2 is close to completion. First ACH produced in laboratory scale! Calcium aluminate slag produced in pilot scale by ELKEM was delivered to Mytilineos in Autumn 2021 and a representative sample was shipped to **NTUA** for leaching work. Acid leaching conditions for optimum Al extraction were determined. At the same time filterability of the pulp is ensured. Some difficulties are encountered still by the presence of intermetallic and metal phases present in the slag, so a pre-oxidation process is being tested before shifting to pilot scale tests later this year.

5 L of the pregnant leaching solution (PLS) was produced under the optimum leaching conditions on a lab scale to also study the precipitation. The crucial parameter concerning precipitation was the endpoint of the experiment to achieve high Al precipitation rates but also have relatively low impurities. A large quantity (≈ 300 g) of aluminium hexahydrate (ACH) was produced under predefined optimum conditions. Over 97% aluminium recovery yield was achieved and over 97% ACH purity. Sampling was performed on the resulting ACH. One sample was kept in NTUA for further analysis, and the rest of the material was shipped to our partner SIQAL to further optimize the purification of the ACH.

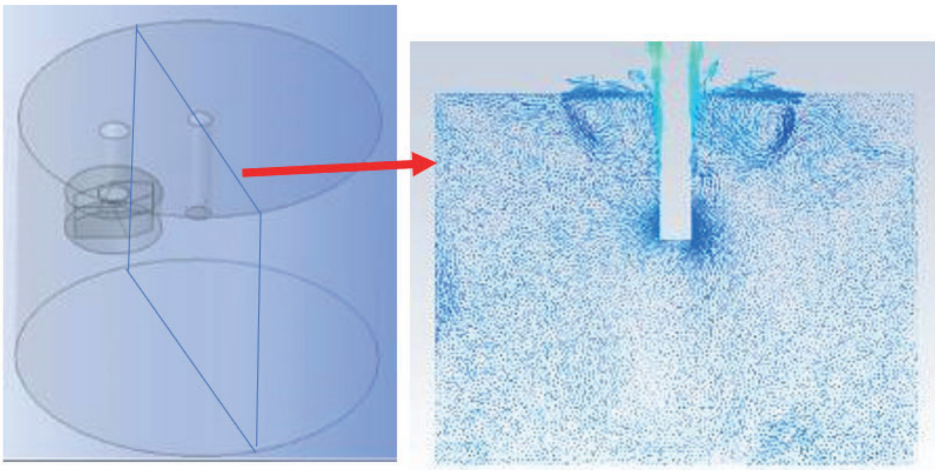
Aluminium bearing solution after the leaching process



Laboratory filtration of the residue proceeds without issues

Hydro has mainly been active in WP1 during the first half of the project and their main activity has been on delivering material to Elkem's pilot trials and NTNUs small scale experiments. Materials that have been delivered are pure aluminium, dross and aluminium scrap. Hydro has also contributed to the resource mapping and to the report about Al-scrap, dross etc that was part of a deliverable in WP1.

CITMAga (formerly ITMATI) performs the numerical simulation of induction and rotary furnaces to model, assess and optimize interactions between physical phenomena. During the second year of the project, the researchers faced new challenges in the modelling of the furnaces such as (i) the multiple steps in the furnace charging strategy, (ii) the interaction of a gas lance to stir the slag, (iii) the mechanical rotor inside the furnace in the stirring process, (iv) ensuring sufficiently fast computer simulations despite their high complexity. As a result of the cooperation, CITMAga enriched the proposed models through the experiment campaigns deployed by FRey and Elkem. So far, input parameters from the experiments allow CITMAga to overcome the lack of references in literature and to take a step forward to validate the computational fluid dynamics models for the SisAl Pilot project. This research aims to make recommendations to predict and optimize the operation of the furnaces, with the purpose that the technological process can be implemented on an industrial scale.



Slag-aluminum velocities in a simulation with gas injection and rotor motion in an induction furnace.

Distribution of temperatures in the FRey rotary furnace.

SINTEF and WP1 partners have finished and submitted deliverable D1.1: Resource mapping report, 1 st. version, and working actively on deliveries D1.3: Report on the final selected raw materials (quartz and lime) properties (particle size distribution, mineralogy) and compositions for different SisAl products, D1.4: Report on Si-slag scull and plant SiO2 fines properties, compositions and D1.5: Report Al scrap, dross, processed alumina and Si-slag properties, compositions and pre-treatments necessary for SisAl process all due by 30. April 2022.

In WP2 a report on Rheological properties of Al2O3–CaO–SiO2 slags has been prepared and published. (Rheological properties of Al2O3–CaO–SiO2 slags - ScienceDirect) SINTEF has also been contributed to the hydrometallurgy pilot in WP3 and will later in the project test produced alumina.

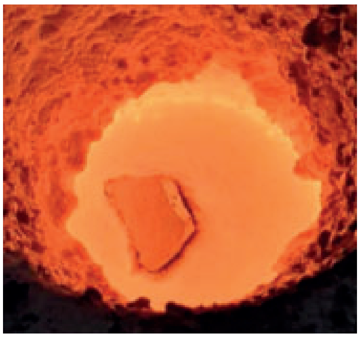
In SisAl Pilot, **SBC** is responsible for the Spanish business case as well as the coordination between the Spanish partners. Focus for SBC has lately been on coordinating and supervising experimental trials at Fundiciones Rey foundry plant. Until now several tests with aluminium dross received from Befesa and quartz received from Erimsa have successfully been done.at. The technical manager of the project Mårten Görnerup together with two persons from ITMATI, who are working in the mathematical model of the induction and rotary furnace, visited the foundry in February and during one of the tests. During the visit they had fruitful discussions about the ongoing and upcoming experimental trials. . The results from the tests are very promising. Silicon has been produced and samples have been sent to the NTNU and to the laboratory in the Santiago University for analysis. In the next couple of months, more tests will be conducted and the whole SisAl team will visit Fundiciones Reyby the end of June in Santiago.

During the last months of the SisAl Pilot activity, Fundiciones REY has started to demonstrate the Pilot 2 process (production of SiAl-alloy) in a 200KW induction furnace facility using a graphite crucible.

Quartz, lime and aluminium mixture have been melted controlling the process temperature, the power of the furnace and the general consumptions. A specific methodology to conduct the Pilot 2 trials has been allocated.

The slag and the silicon have been separated and analysed as well and very interesting and promising results have been achieved.

New trials with different concentrations of raw material are planned for the upcoming months.



The availability, chemical composition and representativeness of certain potential sources of SiO2, namely slag scculls, micro silica and quartz fines as by-product streams of the metallurgical-grade silicon (mg-Si) production operation, were investigated. In this context, particular focus was put on slag sculls (Figure 1). A methodology of sample preparation was developed, and a series of slag scull samples were taken, prepared and analyzed for their composition of matter. The analysis of the sculls shows a large variation of metallic silicon, as well as for other elements. Next to the human factor in the casting process separating silicon from slag (Figure 2), parameters like temperature control during tapping/refining and notable variations in the base composition from ladle-to-ladle play important roles here. Sub-testing of a particular slag scull sample reiterated the inhomogeneity of slag scull material, showing e.g., a variance of factor 2 to 3 for aluminium and calcium content. To get to more consistent results on slag scull composition the mean value of larger quantities could be an attempt. The analytical data of micro silica and quartz fines as by-product streams from **WACKER**´s Holla Metall operation confirm the chemical composition available in the public domain of those materials. In the case of quartz fines the chemical composition resembles the quartz supplied as raw material. The three by-product streams of mg-Si production combined are typically available in several ten thousand tons per such metallurgical site per year.

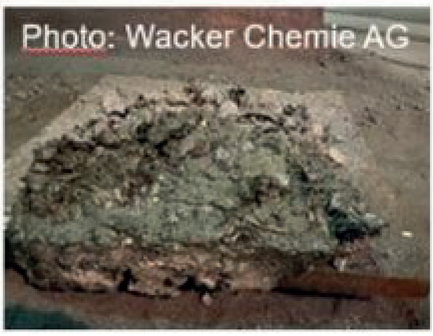


***Figure 1:** Cooled scull sample on ore plate*

***Figure 2:** Casting / emptying of ladle in casting bed*

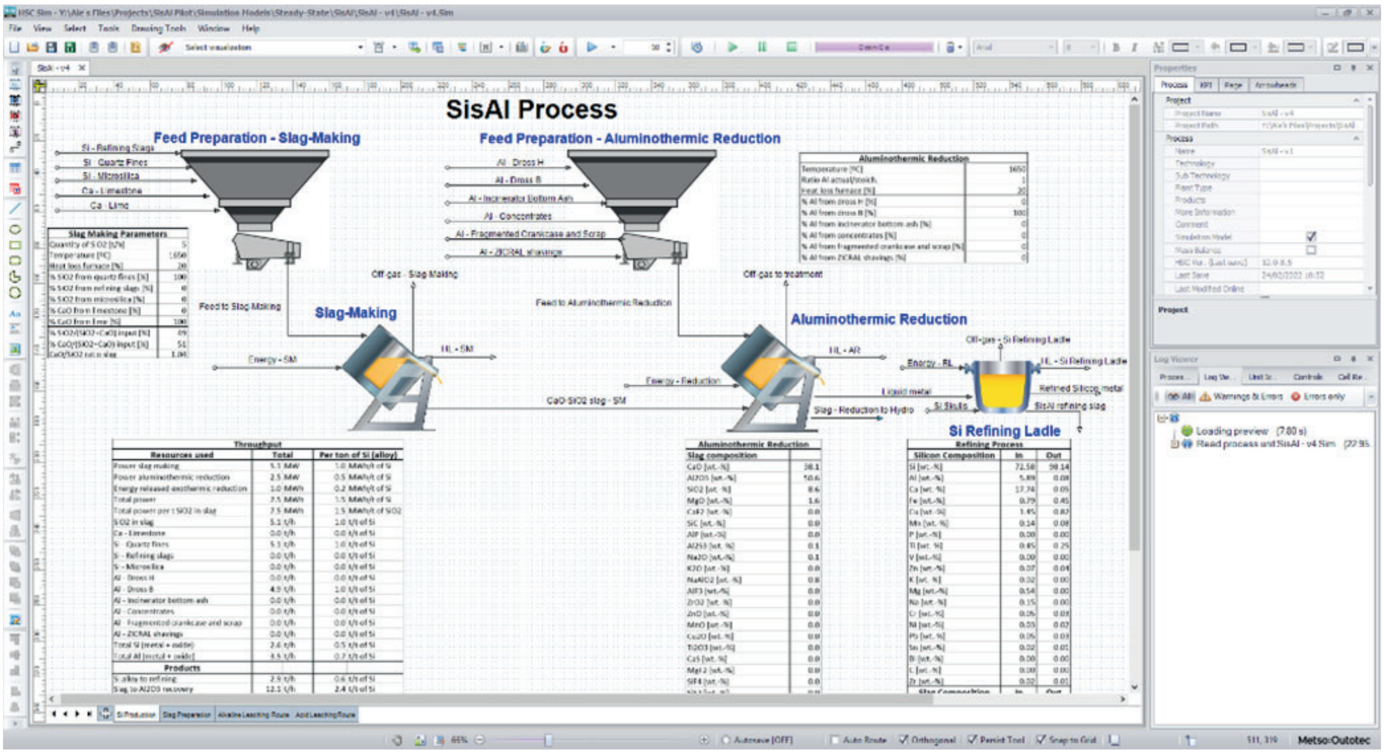
SiQAL performed numerous purification experiments to understand the correlation between quality of the input leaching liquid (PLS) and SiQAL's final product (High Purity Alumina, HPA). In this context impurity segregation and process yields have been studied to finally give a specification for the PLS to allow to make 4N HPA after three or four refinement cycles.

Different routes for the backend of the process, where an intermediate product is converted into alumina have been tested. The resulting products were characterized for purity by GDMS, grain size and morphology by SEM and crystallographic phases by XRD.

Visits at partners sites have been made (NTUA, Mytilineos in Athens) to review and coordinate the joint work.

Dow is both a producer and user of chemical grade silicon, so is excited by the sustainability and economic possibilities offered by SisAl. Together with other consortium partners, we have been collaborating to identify suitable raw materials and assisting with the final pilot furnace design. We are excited to participate in the next scale pilot furnace trials and to learn more about the possibilities of SisAl.

The process simulation models were further developed in the second year of the project. These models consider now all the possible raw material combinations available within the SisAl project (see figure). Accordingly, the models calculate the mass and energy balances of the SisAl process for all the silica and aluminium raw materials combinations. These mass and energy balances have been transferred to the NTNU to perform a LCA of the SisAl process using different raw materials and compare it with the conventional silicon production route from the environmental point of view. Moreover, these mass and energy balances will also be used to benchmark the SisAl process against the conventional silicon production route from the technical, economic and environmental point of view.



Caption of the figure: “Process simulation model for the SisAl process showing the Slag-Making, Aluminothermic Reduction and Silicon Refining steps.”

Minteks pilot campaign is planned in August 2023. A 2 - 3 MW DC arc furnace will be used for Mintek pilot campaign. In the second year of SisAl project Mintek closely monitored and discussed the results obtained from test work conducted at Elkem and NTNU. The information from test work was used as basis for work on process flowsheet for Mintek’s pilot campaign. Mintek used simulation tools like Factsage and Pyrosim to propose various process flowsheet scenarios for pilot campaign.

Extensive, discussions were held between WP2 partners to get inputs and to finalise the process flowsheet for Mintek’s pilot campaign.

Based on the flowsheet, quantity of raw material required for the pilot campaign was estimated. Raw material for **Mintek** pilot will be sourced locally and Mintek has identified the raw material suppliers.

Mintek is continuously engaging with modelling and simulation teams (SIMTEC & HZDR) to simulate the pilot scale operations.

Mintek pilot facility

The purification of metallurgical grade silicon obtained from one of Elkem's pilot trial has been initiated by using the aluminum-silicon melt followed by acid leaching to remove the aluminum.

The first trial was conducted using a small scale sample of 390 [g] of Al + (360 [g] of Si, which is represented by 52% (Al grade 6060) and 48% (MG-Si). The silicon addition to the aluminum melt was in three batches to ensure the homogeneity of the mixture, Figure 1 depicts the melting, mixing, heating, and cooling processes. The 52%(Al)/48%(Si) mixture was heated up to 1100 [°C] and held the temperature for several hours to ensure all the silicon particles are melted, and so, the different contaminations like phosphor, boron, and other contaminations transferred from the silicon into the melt. The mixture was cooled to 850 [°C] and then, the melted aluminum was poured. A decent quantity of the silicon was solidified at this temperature, were, the resulted silicon is still mixed with aluminum.

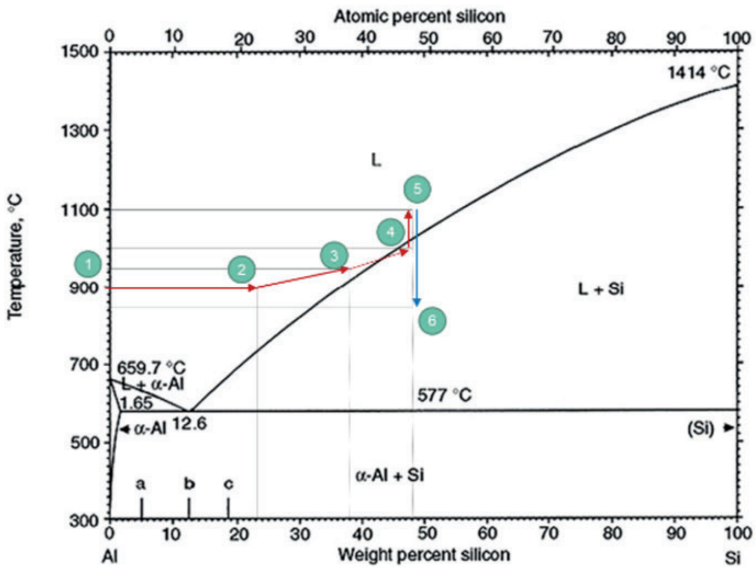


Figure 1

The second step of purification is to remove the aluminum from the solidified Al/Si mixture by means of acid leaching, hydrochloric acid (HCl) is used in this step. before starting the chemical reaction, the solidified mixture was milled into small particles and roasted to ensure a higher surface that will be exposed to the acid. Different acid concentrations and reaction environment temperatures additional to the solid particles to liquid acid ratios are under study to ensure the best conditions that help to get pure silicon.

The particles are washed and filtered to proceed with characterization. Scanning electron microscopy (SEM), Energy dispersive X-ray analysis (EDX), and X-ray diffraction analysis (XRD) are all used to get a better understanding of the results.

In the last 12 months, **Elkem** performed 22 SisAl pilot tests with production of silicon metal and Ca-aluminate slag.

Different aluminium sources were tested: pure aluminium, aluminium scrap and white dross. Results showed yields and compositions in accordance with lab scale testing at NTNU

Furthermore, refining of SisAl produced silicon was performed, using silicon slag and sculls from Wacker. Refined silicon reached a purity of 98,5 % Si.

All tests were completed without any major problems. Furnace operation, raw material charging and tapping performed as expected

The robot for handling hot & heavy operations worked perfectly, and saved operators from manual handling.

Robot in operation

SisAl tapping at Elkem

In the second year of SisAl Pilot, **CiaoTech**, leader of the Dissemination and Communication work package, deployed the communication strategy planned at the project beginning.

The implemented actions aimed at engaging the general public towards the key messages vehiculated in the project, dealing mainly with circularity and significant lower environmental impacts. The dissemination strategy of the project addressed selected stakeholders interested in the fields of aluminium and silicon, spreading the main project results and findings.

All the actions implemented by the partners in the D&C plans were coordinated and supervised to deploy what previously defined, including updating the project materials with the new results achieved.

The project website was always updated with interesting events in line with the project scope as well as with news and updates on progress over the project status; the SisAl Pilot social media channels have been continuously managed to provide information and updates about the project activities and the main D&C outputs were made public also during online events and initiatives virtually attended by the partners. Despite of the spread of COVID19, the project attended also onsite events to display latest project results. Several videos shot during the tests campaign implemented at Elkem and FREY's were published, and SisAl Pilot partners were interviewed on local newspapers and articles were released on the press. Three issues of the project newsletters were released, namely #2 focusing on the first-year results, #3 on the tests and numerical modelling of the SisAl Pilot process, the #4 on second year project results (the newsletter you are now reading 😊 !)

SisAl Pilot managed also joint initiatives such as training courses, with other H2020 projects, available at the following page and advertised also on the website.

The consortium gathered in Aachen in October 2021 for a progress meeting hosted by RWTH, and the next one will be organized in Spain at CITMAga in June 2022.

In the framework of SisAl Pilot project, INNEN is involved in WP6, which is dedicated to dissemination and communication activities, and at the end of this second year the main important and biggest outputs can be listed as follows:

- dissemination kit – 2nd version (available to the link: https://www.sisal-pilot.eu/documents_cat/dissemination)
- updates and maintenance of the website online at the URL: https://www.sisal-pilot.eu
- Promotional video: https://www.youtube.com/watch?v=YK4mKrjT-Qo
- Support to the dissemination activities, in creating helpful cards and materials to promote the project's results

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