



Amorphous Silica Production from Serpentine and its Techno-Economic Analysis

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Abstract: The production of silica from the reaction of serpentine mineral with sulfuric acid has been investigated. Silica production process was developed by using optimum parameters of 67.3 °C temperature, 4.8 M acid concentration and 57.1 min. reaction time. Techno-economic analysis of the silica production process was made using a SuperPro Designer software (Version 9.0). Simulation has been performed for different annual plant capacities between 4,000 t and 16,000 t. The optimum production cost of silica was obtained for an annual plant capacity of 12,000 t. The total capital investment and operating costs for silica production facility designed was calculated as US\$40,247.000 and US\$4.24/kg, respectively. The payback period of the facility investment was determined as 4.8 years. The tailing waste of chrome ore could be used as raw material to produce silica and magnesium sulfate in an economic way.

Keywords: Amorphous Silica, Serpentine, Optimization, SuperPro Designer, Techno- Economic Analysis

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1. INTRODUCTION

Serpentine is a general name used for minerals having the structure of magnesium hydrated silicates. The general composition of those minerals is as following: $Mg_{3-x}(M)_xSi_{2-y}(T)_yO_5(OH)_4$, where the site M could be Mg^{2+} , Fe^{2+} , Fe^{3+} , Al^{3+} , Ni^{2+} , Mn^{2+} , Zn^{2+} and the site T by Si^{4+} , Al^{3+} , Fe^{3+} (Carmignano et al., 2020). The structural configuration of serpentine is shown in Figure 1. It is a layer with a thickness of 0.72 nm in which the Mg trioctahedral sheet $[MgO_2(OH)_4]^{6-}$ is linked to the tetrahedral silicate sheet $[Si_2O_5]^{2-}$ (Carmignano et al., 2020; Fedoročková et al. 2014).

Serpentine is found as chrysotile, lizardite and antigorite in nature. But their crystal structures are different from each other. Serpentine could transparent, white, grey and sometimes green tones, yellow and rose pink in colors. The most widely found serpentine mineral is chrysotile. Although lizardite, antigorite, and chrysotile have the similar chemical composition, serpentines have different layered structures ended up fibrous chrysotile, lamellar agglomerated antigorite and lizardite elongated mineral particles (Zhou et al., 2017; Pietrikova et al., 2004). Serpentine is usually used in specific paint production, fire prevention, paper coating, and vehicle brake systems.

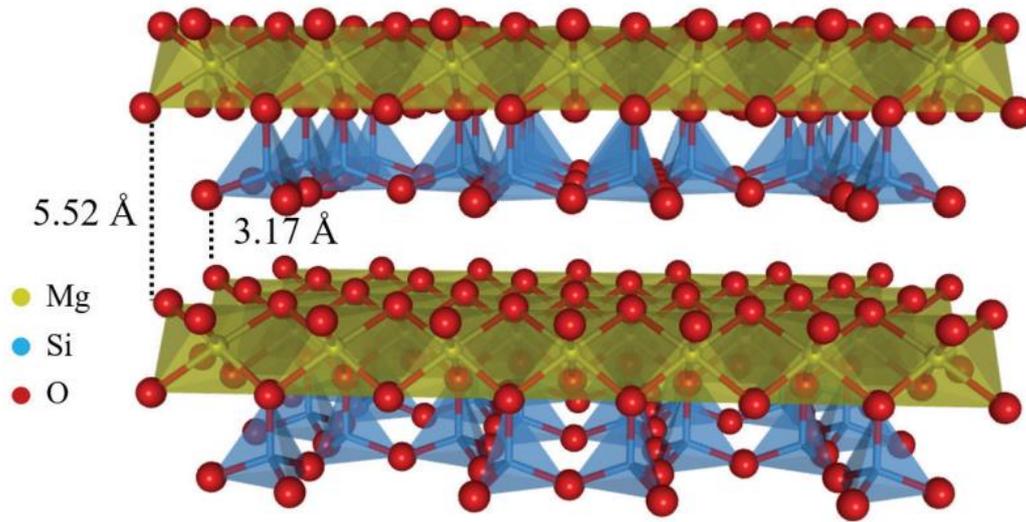
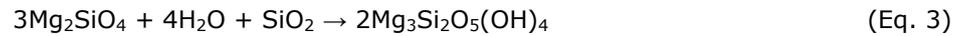


Figure 1. Basic structure of serpentine mineral (Carmignano et al., 2020).

Serpentine minerals are formed by the hydration of olivine minerals which are known as forsterite and fayalite (Mg^{2+} , Fe^{2+}) $_2\text{SiO}_4$ as given in Eqn. 1 to 3. Carbonates and hydroxide are formed as by-products in the following equations (Çevik, 2006). The quality

of dunite mineral is greatly affected during the serpentine formation. The weakened mineral structure enables the dissolution of the mineral in leaching.



Serpentine a silicate mineral which has rich in magnesium and silicon. The structure of serpentine having 35-40%wt. MgO, 30-35%wt. SiO_2 and 15-20%wt. H_2O . However, it has trace amounts of Fe, Al, Ca, Cr, and Ni. Serpentine mineral consists of silicate layers which are connected to magnesium hydroxide $\text{Mg}(\text{OH})_2$ by weak ionic bonds (Fedoročková et al., 2014). Serpentine minerals are easily dissolved due to the presence of these ionic bonds. Serpentine minerals are formed as concentrated waste of chromium production plant in Türkiye. The abundance of serpentine in Earth's crust makes it is cheap and accessible.

precipitated silica is US\$ 3.8 Bn in 2022 and estimated to increase with rate of 4.4% from 2023 to 2031 (Global Industry Report, 2023). The important producers of silica in the world are Evonik Industries, PPG Industries, Wacker Chemie AG, AkzoNobel N.V., Tosoh Corporation, Cabot Corporation, and Solvay SA.

Silicon is the second most abundant element which are found as compounds after oxygen. The 25.7% of earth crust consist of silicon compounds (Norton, 1993). SiO_2 , silicon dioxide, or widely known as silicate in industry make bond with metals (magnesium, aluminum, calcium and iron) to form magmatic and metamorphic rocks and silicate minerals (Flörke et al., 2000). Silica has many polymorphic structures such as crystalline silica, amorphous silica and synthetic amorphous silica. Crystalline silica can be found in nature as silicone dioxide and the most encountered type is quartz. Amorphous silica means that it has no crystalline unit in its structure. Synthetic amorphous silica can be produced via different techniques from silica sources. The use of synthetic amorphous silica has been increasing each year in the world. The global industry analysis reported that the worldwide demand for

Hereinafter, the term "silica" will be used instead of "amorphous precipitated silica" in the text. Silica is a white powder having the density of 1.9-2.2 $\text{g}\cdot\text{cm}^{-3}$. It has SiO_2 content of 95%wt or greater. The surface of area of silica varies between 50-400 $\text{m}^2\cdot\text{g}^{-1}$ which enables it to be used as filling materials (Florke et al., 2000). Silica produced is named according to the specific technique utilized, such as precipitated silica and pyrogenic silica (Lazaro et al., 2012). There are two main methods utilized in silica production: wet method and thermal technique. Pyrogenic silica is obtained in the gas phase reaction of tetrachlorosilane and hydrogen at an elevated temperature. HCl gas formed as by-product is separated by adsorption from the process to be reused in the production of SiCl_4 (Flörke et al., 2000). Wet method consists of two steps: first steps is the synthesis of sodium silicate solution via the reaction between olivine and NaOH/KOH at high pressure and temperature (120-200 °C) in an autoclave and the second step is the reaction of sodium silicate with acidic solution to give precipitated silica as product (Raza et al., 2018). Commercially sodium silicate known as water glass is directly used in silica

production. When the process is evaluated overall, due to the high energy consumption and CO₂ emissions it is not sustainable and environmentally friendly.

Precipitated silica can also be produced from natural silicate minerals like olivine, serpentine using the wet process. The production of precipitated silica from olivine mineral was studied by Lazaro and his co-workers (Lazaro et al., 2019). Olivine mineral having particle size range of 0.1-0.5 mm and of 0.2-1.2 mm was dissolved by using 3 M H₂SO₄ at 50 °C for 24 h. A high quality ordered mesoporous silica production in a one-step synthesis with the use a micellar liquid crystal prepared from the non-ionic surfactant Triton X-100 was investigated. The produced mesoporous silica has the surface area up to 1000 m²/g and narrow pore size distributions around 2-4 nm (Lazaro et al., 2019). The production of Mg(OH)₂ and MgO was investigated using serpentine mineral (Ballhorn and Franke, 1996). As the serpentine has less stable structure, it is easily digested by acids. The reaction between serpentine and sulfuric acid produces precipitated silica and sulfate salts as by-products depending on the impurity content of the mineral.

Silicic acid is initially formed in an aqueous phase during acid dissolution of silicate minerals. Silicic acid monomers form oligomers and polymerize to form larger units at certain conditions pointed by Iler (1979). Silicic acid can be produced in both acidic and basic solutions. In acidic medium, viscosity of the mixture increases depending on polymerization degree of Si(OH)₄ units. Aggregation and flocculation occur in an acidic medium regardless of charge balance. As the reaction progresses, released SiO₂ from mineral structure hydrolyzes to form silica polymeric units and silica gels. In the production of silica from minerals by using acids, product should be separated from the reaction media as soon as possible to prevent the gel formation. Recovery of Mg from H₂SO₄ leaching solution of serpentine was studied Chen and his coworkers (2023). Fe³⁺, Al³⁺, and Cr³⁺ in solution were precipitated increasing pH to 4 by adding MgO. After this step, 6 g/L Na₂S solution was added to the filtrate and reacted for 1.0 h to remove Ni²⁺, Co²⁺, and Mn²⁺ in the form of NiS, MnS, and CoS, respectively. Finally, diluted solution was mixed with ammonia to precipitate Mg(OH)₂ at a temperature range of 40-70 °C; and the pregnant solution was further mixed with NH₄HCO₃ to precipitate (4 MgCO₃.Mg(OH)₂.4H₂O). The purity of Mg(OH)₂ was reported as 98.48% (Chen et al., 2023).

Production of potassium-magnesium chloride from the chromite ores concentration plant tailings was studied in the literature. Chromite ores concentration plant tailings used as Mg source was firstly threatened by wet magnetic separation. Concentrated raw material was dissolved by using concentrated HCl to produce magnesium chloride and amorphous silica. The purity of amorphous silica was reported as 95.75%wt. To produce purer magnesium chloride, Mg(OH)₂ was added into solution to increase pH so that impurities can precipitate. Finally, KOH

was added stoichiometrically into the purified MgCl₂ solution at 90 °C to produce carnallite (Top and Yıldırım, 2017). In most of the studies in literature, temperature, acid type, acid concentration and reaction time have been investigated and optimized (Pietriková et.al., 2004; Çiftçi et al., 2020). There is no information about the techno-economic analysis of the developed process.

SuperPro Designer is a software which enables the modelling and simulation of the developed process to determine its feasibility. The process can be designed either in batch or continuous mode. The required data e.g. physical and chemical properties of raw materials and products, economic parameters, reaction parameters and utilities for the process can be selected from its database if available, or experimentally obtained from lab studies and from the market. After designing the process, mass and energy balances are calculated simulations are performed. The feasibility of boric acid production from colemanite mineral was investigated by using SuperPro Designer. The fixed capital investment and annual operating costs of the boric acid plant are calculated as US\$170.7 M and US\$70.1 M/y, respectively, for a 100 kt/y capacity. Boric acid production cost with the impurity of <100 ppm was calculated as US\$0.70 kg⁻¹. A 6.3 years of payback time was estimated for the developed process considering environmental effects (Gönen et al., 2022).

The feasibility analysis for silica (SiO₂) production from the reaction of serpentine mineral and sulfuric acid has been aimed in this study. Silica production process was formed in SuperPro Designer software, version 9.0 (Intelligent Inc., USA). Mass and energy balances were made for the developed process for different plant capacities. Payback periods (PBP) for those capacities were estimated from the simulation results.

2. EXPERIMENTAL SECTION

2.1. Materials

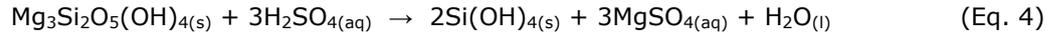
Serpentine mineral is an inert powder formed as a waste from the processing of chromite ores concentration plant tailings in Bursa, Türkiye. The serpentine mineral used in the experiments was supplied as -150 µm from Hayri Ögelman Inc. In the experiments, sulfuric acid was used to digest the serpentine mineral to produce an amorphous silica. The sulfuric acid used in the experiments is 98%wt. Chemical analysis of the serpentine mineral used in the experiments was carried out using X-Ray Fluorescence.

2.2. Methods

In the production of silica from the reaction of serpentine mineral with sulfuric acid, solid/liquid ratio: 10%, stirring rate: 1000 rpm and particle size of serpentine mineral: -150 µm were kept constant. Temperature range of 50-70-90 °C, the acid concentration of 3.0-4.0-5.0 M and the reaction time of 20-40-60 min. were chosen as experimental parameters and optimized in the previous study

(Çakan and Gönen, 2022). Those optimized parameters were used in the process design and development in SuperPro Designer program. The heterogenous reaction between serpentine and

sulfuric acid in an aqueous phase was carried out in a stirred glass reactor as given in Eq. 4.



The solid phase formed in the reaction was silica and the liquid phase containing the magnesium sulfate. Vacuum filtration method was used to separate the solid phase from the solution. The amorphous silica obtained in the filtration was washed with distilled water to purify and dried in an oven at 105 °C. X-Ray Fluorescence analysis was performed for determining the chemical composition of silica. FTIR analysis was performed for the bond structure of silica. Conversion in the reaction was determined by using the Mg concentration measured by ICP-OES analysis. A 4.8 M acid concentration, 67.3 °C temperature and 57.1 min reaction time were determined as the optimum conditions of the reaction.

2.3. Economic Analysis

Those optimized parameters were entered as a data into the SuperPro Designer program. Silica production process was designed in a continuous mode. After the continuous operation of the process is determined, the raw materials, by-products,

utilities and products used in the whole process were defined in the program. Then equipment to be used in the process were selected. The silica production process consists of a continuously stirred tank reactor (CSTR), belt filtration, washing unit, drying unit and neutralization tank. The acid concentration of 4.8 M, reaction temperature of 67.3 °C and the reaction time of 57.1 min. were used as a reaction parameter in the SuperPro Designer program. The stoichiometric reaction was selected in the reactor. The reaction mixture at the end reaction was fed to the filtration unit. The amount of solid to be separated in the filtration unit was determined from the experimental results and used in the program. After the filtration unit, silica is fed to the washing units where soluble MgSO_4 salt was removed. Finally wet silica was dried in drying unit. The magnesium solution obtained from filtration was neutralized with ammonia to obtain magnesium sulfate and ammonium sulfate solution. The flow diagram drawn with the SuperPro Designer program in line with these data is shown Figure 2.

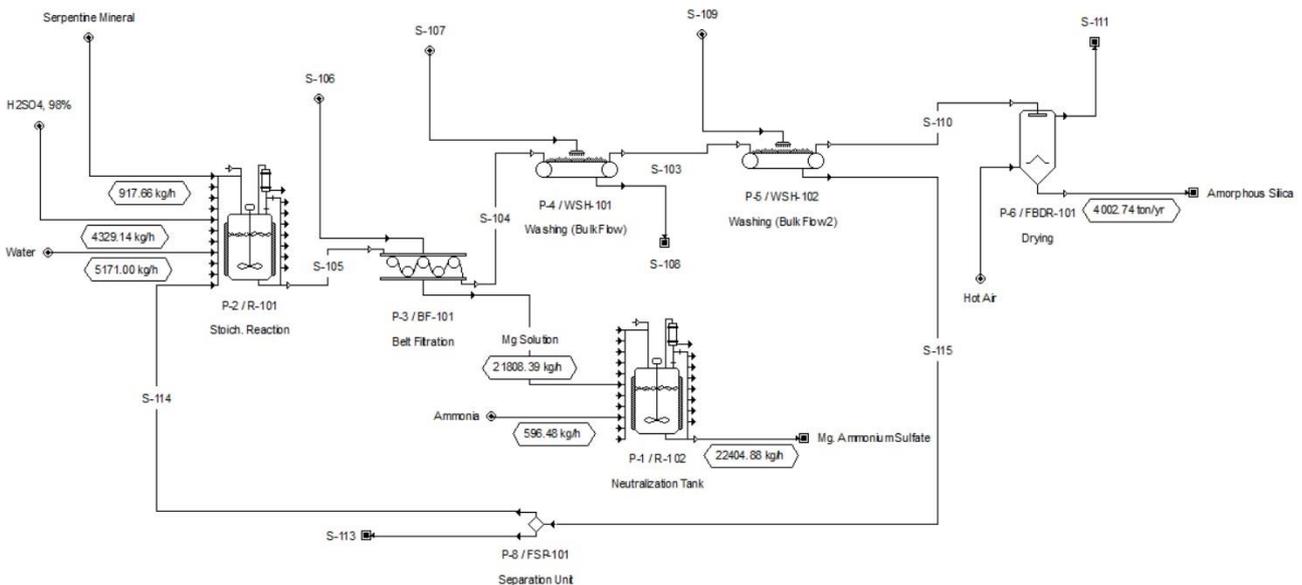


Figure 2: Flow diagram of the silica production process.

The economic analysis of the proposed facility was carried out by evaluating the facility capacity and purchase costs and production costs of the serpentine mineral. As seen in Table 1, total capital investment is obtained by summing total direct cost (TDC), total indirect cost (TIC), contingency (CFC), working capital and startup costs. For direct cost (DC), it consists of piping, facility construction, equipment cost, installation, and other related costs. Indirect costs consist of engineering and construction-related expenses. The economic parameters (e.g., purchasal cost of raw materials and other consumables and

selling price of products) were obtained from the market.

The total production cost was estimated from the mass and energy balances, prices of raw materials, chemicals, labor cost, and utilities as shown in Table 2. Transportation cost of raw materials and products to the plant and market was excluded from the production cost calculation, as it is assumed that the plant was built in close to the mining site. Labor and utility costs parameters were selected from the software database as given in Table 2. Reactor design parameters e.g., residence time, reaction

temperature, conversion was obtained from experimental studies (Çakan, 2022). The solubility of magnesium sulfate was taken from the literature (Chen et al., 2023; Carmignano et al., 2020). A straight-line method was used for depreciation calculations in which a 10-year period and a salvage value of 5% of the equipment purchase price were considered (Turton; et al. 2009). The feasibility of the

suggested process is determined using the payback time (PBT) equation, which is given in Eqn. 5.

$$\text{Payback Time (years)} = \frac{\text{Total Investment}}{\text{Net Profit}} \quad (\text{Eq. 5})$$

Table 1: Total capital investment (US\$M) parameters and results for silica production process.

Items	Estimation approach	Plant capacity (t./y)			
		4000	8000	12000	16000
Total Direct Cost (TDC)					
Equipment purchase cost (PC)	Listed equipment cost	3.33	4.21	5.84	7.79
Installation	Equipment specific	1.22	1.60	2.15	2.89
Process piping	0.2 x PC	1.17	1.47	2.04	2.72
Instrumentation	0.1 x PC	1.33	1.69	2.34	3.12
Insulation	0.03 x PC	0.10	0.12	0.17	0.23
Electricals	0.1 x PC	0.33	0.42	0.58	0.78
Buildings	0.05 x PC	1.50	1.90	2.63	3.50
Yard improvement	0.1 x PC	0.50	0.63	0.87	1.17
Service facilities	0.2 x PC	1.33	1.69	2.34	3.12
Total Indirect Cost (TIC)					
Engineering and supervision	0.25 x TDC	2.70	3.43	4.74	6.34
Construction expenses	0.35 x TDC	3.79	4.81	6.64	8.87
Contractor's fee & Contingency (CFC)					
Contractor's fee	0.05 x (TDC + TIC)	0.86	1.10	1.52	2.03
Contingency	0.1 x (TDC + TIC)	1.73	2.20	3.04	4.05
Working Capital		1.19	2.36	3.54	4.72
Startup cost		1.00	1.26	1.74	2.33
Total Capital Investment (TCI = TDC + TIC + CFC)		22.11	28.91	40.25	53.69

Table 2. Operating cost parameters.

Items	Estimation Approach & Assumption
Total variable production costs	
Raw materials	From mass balance
Labor- dependent	Annual salary: 20,000 US\$ Supervision factor: 0.15
Utility cost	Electricity: 0.10 US\$/kWh Steam: 12 US\$/MT Stream (high pressure) 20 US\$/MT Process water: 0.12 US\$/m ³ Cooling water: 0.05 US\$/m ³ Chilled water: 0.4 US\$/m ³
Maintenance & repair for Equipments	7% of the TCI
Laboratory cost for QC and QA	15% of the total labor cost
Operating supplies	15% of the equipment maintenance and repair cost
Royalties	4% of the capital cost
Fixed Charges	
Depreciation (10 year straight line)	Depreciated 5% of the TCI
Insurance	1% of the TCI
Plant overhead costs	50% of labor, equipment maintenance and repair cost
General Expenses	
Administrative costs	15% of operating labor cost
Distribution and marketing costs	No less than 2% of the total operating cost

3. RESULTS AND DISCUSSION

Serpentine mineral $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ is a good source of Mg and Si. Especially, serpentine is produced as tailing waste of chromite concentration plants in

Türkiye. The utilization of these waste mineral for production of magnesium products, such as $MgSO_4$ and $Mg(OH)_2$; and precipitated silica can be economic and feasible. Another issue is that the quantity of tailings has been increased with respect to chrome ore production in Türkiye (Top and Yıldırım, 2017).

Precipitated silica could be produced from both olivine and serpentine minerals. As the serpentine is more labile than olivine mineral based on the structural configuration, serpentine mineral is easily digested by acids. Secondly, as serpentine is obtained as a tailing waste during the chrome ore concentration, there is no need to extra mining processing. Because of those issues, serpentine mineral obtained from Ögelman Mining Inc., was used as raw material to produce silica and magnesium sulfate solution. The techno-economic analysis of silica production from serpentine mineral was performed using SuperPro Designer software.

3.1. Total Capital Investment

Technical and economic investigations of the silica production process from serpentine mineral were examined for different plant capacities. It allows the determination of annual operating cost (AOC), total capital investment (TCI) and unit production cost of amorphous silica. The necessary parameters in determining the technical analysis for the entire project were made using 2022 data. The construction of the facility, simulated with SuperPro Designer, started in 2022 and completion is expected to take 30 months. The project life is determined as 15 years, the number of annual working days is 330 days and the commissioning period is 4 months. Net present value (NPV) is calculated using 4% interest rate. The amortization of the capital investment is assumed to be 10 years and the initial cost of the salvage fee is assumed to be 5% of the equipment initial cost (Peters and Timmerhaus, 1991).

The total capital investment (TCI) equipment purchasing costs of the silica production facility were determined from the database of the program, and the costs of products and raw materials were determined from the global market prices. The size of the equipment and the number of equipment required for the capacity were calculated with the defined mass and energy balances. Care was taken to select stainless equipment suitable for the sulfuric acid used to dissolve the mineral in the reaction. The total capital investment of the amorphous silica production process is between US\$ 22.11 M and US\$ 53.69 M, depending on the capacity of the facility (4–16 kt/year), as shown in Table 2. In addition, it can be seen in Table 2 that the investment cost increases

depending on the production amount in chemical plants. According to the examined facility capacities, the payback period of the investment was determined as 5.3 years for 4,000 t/year. It was observed that by increasing the facility capacity by 12,000 t/year, the payback period decreased to 4.8 years. It was observed that there was no change in the payback period because of further increasing the facility's capacity. For this reason, 12,000 t/year was considered in the technical and economic analysis. The total capital investment required for the annual production of 12,000 t of amorphous silica was determined to be US\$ 40.25 M.

Increasing the capacity of the amorphous silica production facility had a direct impact on the number of equipment. Depending on the capacity, the purchasing costs of the necessary process units have increased. Total direct cost (TDC) is obtained by adding up the installation, process piping, instrumentation, insulation, electricity, buildings, facility construction, yard improvement and service facilities. These items are estimated by multiplying the equipment purchase cost by the coefficients specified in Table 1. For example, process piping is seen as 0.2 times the cost of purchasing equipment. The process piping cost was determined as US\$1.17 M and US\$2.72 M. for production capacity of 4,000 t/year and 16,000 t/year, respectively. After determining the total direct cost (TDC), total indirect costs (TIC) were calculated. Total indirect costs, engineering, and supervision are estimated at 0.25 times the total direct costs. Total non-direct costs were determined as US\$ 6.49 M at a production capacity of 4,000 t/year. Total indirect costs were determined as US\$ 15.21 M. at a production capacity of 16,000 t/year. The total direct cost and non-direct cost varied in the same way as the contractor's fee and contingency (CFC). Total capital investment (TCI) was determined by the sum of these variables. In the study, US\$ 22.11M, US\$ 28.91M, US\$ 40.25M. and US\$ 53.69M TCI were obtained in the production capacities examined as 4,000 t/year, 8,000 t/year, 12,000 t/year and 16,000 t/year, respectively.

Figure 3 represents the variation of both total capital investment and production cost with annual plant capacity ranging from 4,000 t to 16,000 t. As the plant capacity is increased the investment cost increased as well, which is common for the most chemical plants. However, a decrease in production cost was observed when plant production capacity has been risen. The minimum production cost was determined as US\$ 4.24/kg for the plant capacity of 12,000 t/yr.

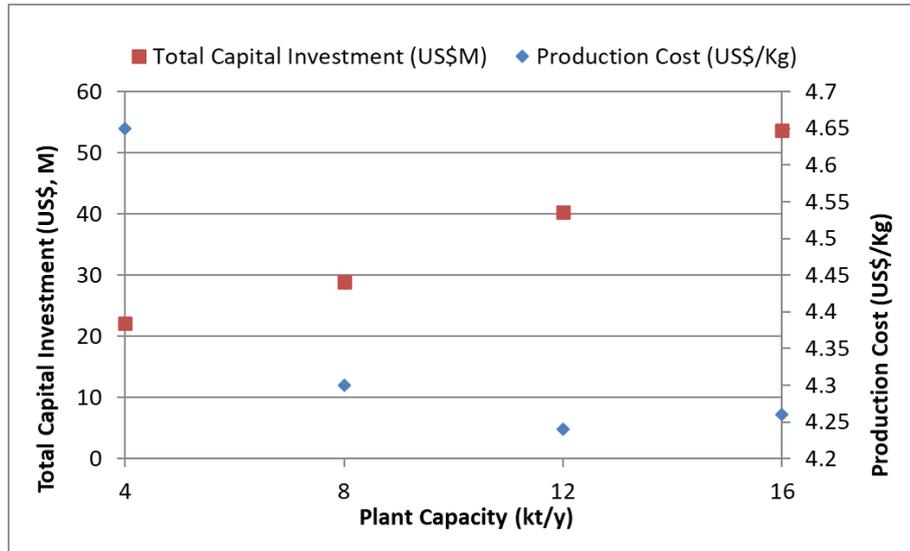


Figure 3: The effect of plant capacity on total capital investment and production costs.

On the other hand, payback time is a good indicator whether the investment is a good choice or not. For this reason, payback time was calculated for each plant capacity and given in Table 3. The minimum

payback time was obtained for 12,000 t annual silica production capacity. If there is a market demand for the silica where the plant would be constructed, this value is remarkable.

Table 3: Payback period of investment according to the plant capacity.

Plant capacity (t./y)	4000	8000	12000	16000
Payback Time (y)	5.32	5.20	4.77	5.86

3.2. Annual Operating Cost

Annual operating cost (AOC) was calculated as US\$45,588,000 for an amorphous silica production capacity of 12,000 t. Raw material cost was determined as the most important expense with 84.4% of the total cost of the facility. When the raw material cost was examined, it was seen that it the highest cost due to the price of sulfuric acid. The purchase price of sulfuric acid and serpentine was entered to the software as US\$ 300/t and US\$ 20/t, respectively. The second most important expense of the facility is facility maintenance, local taxes, insurance, equipment depreciation and other general expenses with 14.4%.

The silica production cost for annual plant capacity of 12,000 t using a purchasing cost of 20 US\$/t of serpentine mineral, was determined as US\$ 4.24/kg. The unit production income of the facility is calculated as US\$ 5.03/kg. The most important factors on which the amorphous silica production cost depends are raw materials (84.4%) and facility expenses (14.4%). In the amorphous silica production facility from the serpentine mineral, the purchase price of sulfuric acid constitutes a significant part of the raw material cost. Sulfuric acid (98%) is sold in the market between US\$ 250/t – US\$ 350/t. The purchase price of sulfuric acid was entered into the program as US\$ 300/t.

In the silica process, the production rates of magnesium sulfate solution and amorphous silica are determined as 407,963 t/year and 11,855 t/year, respectively. The revenue prices of these products

were entered into the program as US\$0.06/kg and US\$2.57/kg, respectively. In line with these data, the revenues of magnesium sulfate were calculated as US\$ 24.48 M and the revenues of amorphous silica was calculated as US\$29.65 M. The total revenue of the facility was determined as US\$ 54.12 M. Annual operating cost (AOC) is determined as US\$ 45.59 M/year. The gross profit of the facility is calculated as US\$ 8.53 M/year, taxes (40%) are US\$3.4 M/year, and the net profit is US\$ 8.44 M/year.

4. CONCLUSION

Serpentine mineral occurring as tailing waste in chrome ore mining was utilized for silica production. The feasibility analysis of the silica production process from serpentine mineral and sulfuric acid in an aqueous phase was performed from mass and energy balances calculated through modeling of the developed process at different annual capacities between 4,000 t - 16,000 t. The total capital investment for the silica production process was determined between US\$ 22.11 M - US\$ 53.69 M depending on the facility capacity ranging from 4,000 t/year to 16,000 t/year. The production cost of silica was determined as US\$4.24/kg for 12,000 t plant capacity. Total capital cost was determined to be US\$40,247,000, annual operating cost was US\$45,588,000, and total annual revenue for main product was US\$54,122,000. The payback period for 12,000 t/year amorphous silica production capacity was calculated as 4.8 years which is feasible value.

5. CONFLICT OF INTEREST

No potential conflict of interest was reported by the authors.

6. ACKNOWLEDGMENTS

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