SOCIETY OF PHYSICS STUDENTS
An organization of the American Institute of Physics

SPS Chapter Research Award Proposal

Project Proposal Title	Feasibility Studies of Dolomites from Phosphatic Pebble for Thermochemical Energy Storage and CO2 Sequestration
Name of School	Florida Polytechnic University
SPS Chapter Number	2054
Total Amount Requested	$2,000.00

Abstract

The current proposal addresses and aims to deploy the highly abundant, uneconomical dolomites (calcium-magnesium carbonates) for the end-use and green technology applications namely, (i) thermochemical energy storage and (ii) carbon dioxide sequestration and capture in fossil fuel power plants. An innovative concept of thermochemical energy storage system and CO_2 sequestration assembly by using the dolomites ($CaO/CaCO_3$ and $MgO/MgCO_3$ reactions) in phosphatic pebble matrices is proposed. The motivation behind the project is that the Central Florida's phosphate mining produces dolomites which are currently piled up in clay ponds and the proposed study is attempt to establish feasibility study to utilize dolomites for thermochemical energy storage and sequester/capture CO_2 in power plants.

Proposal Statement

Overview of Proposed Project

1. Statement of Source and Magnitude of Problem
   The 1914 US Geological Survey Bulletin authored by Van Kauwenbergh et. al, [1] reported the detailed study of the minerology and chemistry of the phosphates mined in Florida, especially Polk and Hillsborough counties of Central Florida. From this report, it is understood, the most uneconomical ingredient in the phosphate matrix is the Magnesium-Calcium Oxides, so called "dolomites". The mining industry often terminates their mining process, when they encounter the dolomites or "dolostones", which are the major source of magnesium and are not much favorable for the fertilizer processing. The dolomites are not only difficult to remove from the clay matrix but also poses "environmental sustainability" issues due to their deposition in clay settling ponds around these counties. The magnitude of this problem is considered to be multifold if the future mining processes continue growing to other counties such as Manatee, Hardee and DeSoto in the State of Florida. The problem of dolomite sedimentation (or stockpiling) in mining plants or clay settling ponds is unavoidable if there is a depletion of mostly wanted "overburden" soil or economic phosphate ores which are the main source of the Phosphorous and Phosphorites for the fertilizer industry.

The presence of the most troublesome magnesium oxide (MgO) contained dolomites are major hindrance factor in the manufacturing of "phosphoric acid" for di-ammonium phosphate (DAP) fertilizer production, since high magnesium content increases the sulfuric acid consumption, and therefore causes its lower production rate and the phosphoric acid yield [2, 3] [Source: FIPR 2011-2016 strategic plan; Energy and Environmental Profile of the US Mining Industry].

A very recent publication by American Institute of Physics where Konstantinos Kakosimos et. al, studied the characterization of surface carbonates as obtained from various regions of Qatar peninsula and have shown promising potential for CO_2 capture and thermochemical energy storage [4]. According to this report dolomites have higher performance in terms of CO_2 sorption and storage capacity when compared to calcite. Long term cyclic study up to 27% thermal energy storage was demonstrated by Judith Perlinger et al in groundwater aquifer, which was plausible by the presence of minerals (dolomites) [5]. Based on the aforementioned factual reports, it is proposed to examine and conduct the feasibility study to utilize the high abundance of dolomites in phosphatic clay matrices for most sustainable and green technological applications such as (i) thermochemical energy storage (TCES) in concentrated solar (CSP) power plants and (ii) carbon dioxide sequestration-capture (Carbon Capture and Storage, CCS) at the fossil fuel base power plants.

2. Impact if Specific Objectives Are Met
   On one fold, the so called uneconomical dolomites per the phosphate industrial waste (termed as "CRISIS"), will create an OPPORTUNITY for the sustainable and green energy applications, for example, a resource sorbents for CO_2 capture in fossil fuel power plants and for thermo-chemical energy storage in packed beds for concentrated solar power plants. If the specific objectives of this feasibility study are MET, then it will impact positively, the FIPR's and its parent institution, Florida Polytechnic University's on-going strategic goals, in creating research based sustainable energy curriculum, creation of thesis or dissertation problem (project) to attract graduate and undergraduate students, commissioning of new Chemical Engineering major program and international graduate program on Green Chemistry/Engineering and Technologies. In addition to state-of-the-art research endeavor, the proposed study will impact on public health safety, and offer K-12 education and outreach opportunities.

3. Benefits of the Proposed Study (SPS Program)
   Demonstrating and deploying the much abandoned "dolostones" or "dolomites" for Green Energy Technologies such as carbon dioxide sequestration-capture and thermochemical energy storage, if the proposed objectives are (nearly) MET, will have potential short term impacts to the economy of State of Florida (in particular Central Florida) and to the United States. The long term impacts in utilizing non-toxic, inexpensive, and earth abundant dolomites for Green Energy applications will reduce the atmospheric carbon foot prints (Green House Gas emission reductions) on our fragile planet "Earth". A successful utilization of dolomites will stimulate interest and early adoption of this material and technology by CSP and conventional power generation industry. This will ensure competitive edge, secure job creation and sustainable prosperity of the local community. Extraction, cleaning and transport of dolomite and subsequent design, fabrication and operation of plant scale TCES and CCS systems will lead to creation of many direct and indirect jobs. In addition, it will create tangible benefits in the form of business attraction, income and revenue growth for the state economy. The SPS undergraduate students of Florida Polytech chapter will greatly benefited by getting research training to work the proposed project and will certainly motivate them to carry out research experience at the undergraduate level and prepare them for graduate studies or workforce in industries or academia.

Background for Proposed Project

4. Review of Pertinent Literature and Related Work in Progress by Others

According to the Intergovernmental Panel on Climate Change, IPCC's report [6], the reduction of Green House Gas (GHG) emissions can be achieved by two means, one way is to sequester and capture the atmospheric CO_2 or coal power plants as-produced carbon dioxide, the other pathway is to develop thermochemical energy storage using earth abundant materials. For both of these applications, it is proposed to evaluate the feasibilities of employing the dolomites or other related carbonates which are abundantly available at the Central Florida's Phosphate mining facilities as the potential TCES/CCS candidate. Dolomites (or dolostones) and limestones are widely used as inexpensive solid sorbents for absorbing CO_2 via sequential calcination and carbonation loop cycles at elevated temperatures.

A comprehensive review of calcination and carbonation of limestone during thermal cycling for CO_2 sequestration was reported by Stanmore and Gilot in 2005 [7]. Overall, this review literature concluded that the prolonged residence times in fluidized bed systems affect the cycling performances of calcite and dolomites. Drupatie Latchman, a graduate student (mentored by Dr. Sesha Srinivasan, investigator of this current proposal) of the University of South Florida's Clean Energy Research Center did her preliminary studies on the CO_2 capture from fossil fuel power plants using dolomite and partially concluded that dolomites immobilized on yttria fabrics lead to increase in CO_2 absorption capacity [8]. Long term calcination/carbonation cycling were reported by Zhongxiang Chen et. al, [9] which demonstrated that the cycling stability of Strassburg limestone and Arctic dolomite increases if these materials are pre-treated for 24h calcination periods. Ke Wang et. al, very recently reported that there is an enhancement of activity and stability in CO_2 absorption, if one can coat MgO by carbon derived from the citric acid. The carbon coating is favorable in achieving smaller grains, larger specific surface area and pore volume while obtaining more uniform distribution of CaO and MgO [10]. In the above study, the authors examined the natural dolomite obtained from Anhui Province of China with compositions of CaO-37.01%, MgO-22.50%, SiO_2-0.11%, Fe_2O_3-0.1%, and CO_2 balance. Another natural mineral dolomite from Seljelid, Norway was used as a precursor to modify Ca:Mg ratio and loaded with nominal compositions of Zr (up to 1wt%). The modified ore has shown superior cyclic performance when compared to the pristine dolomite [11]. The BET specific surface area and pore volume were increased and showed enhance gas sorption kinetics for those pre-treated and Zr modified samples.

Cheap, efficient and non-toxic energy storage technologies are vital to handle the rapidly increasing usage of renewable energy at the energy production sites, for example, grid stations, solar power plants and wind mills. The United States Patent Office issued many patents for the design and development of heat storage units involving calcium hydroxide/dolomite mixtures [USPTO, 4,447,347, Goldfarb et al, May 8. 1984; USPTO, 2,380,480, Werner Syx et al, July 31, 1945] [12, 13]. Solar energy based power generation in concentrated solar power (CSP) plants often deploy sensible or phase change materials (PCM) systems for storing the latent heat of the sun either received in parabolic troughs or dish type configurations. Tanvir Alam's doctoral thesis from USF's Clean Energy Research Center (mentored by Dr. Jaspreet Dhau, investigator of this current proposal) [14] and their breakthrough discovery of encapsulated PCM's reported in 2016 ASME Conference Proceedings on Renewables, thus demonstrated the exergy to energy ratio up to 90% in a packed bed system [15] design. Another important method of storing heat is by thermochemical reactions using calcite and dolomite which undergoes reversible reactions of CO_2 cycling at elevated temperatures. The added advantage of using $CaO/MgO/CO_2$ for such applications can not only store the large amounts of energy per unit mass but it also can upgrade the temperature of the heat energy. The highest thermal CO_2 conversion efficiency (above 50%) is reported in these dolomite systems by upgrading heat at 773K to temperatures above 1073K [16]. Beatriz Sarrion et. al, have very recently demonstrated that the Calcium Looping (CaL) process based on multicycle carbonation/calcination of natural limestone/dolomite (obtained from Bueres, Spain) for thermochemical storage of CSP [17]. Magnesium based layered hydroxide salts have been synthesized and evaluated for thermochemical energy storage by Yamashita et al [18]. In this investigation, the authors have systematically studied effect of several experimental conditions on the synthesis of Mg based layered hydroxide salts by simple aqueous solution derive methods, and their potentials of the multistep chemical heat storage property.

Expected Results

5. Enumeration of the Specific Project Goals

An innovative concept of using dolomites ($CaO/CaCO_3$ and $MgO/MgCO_3$ reactions) in phosphatic clay matrices for the thermochemical energy storage (TCES) system and CO_2 sequestration assembly is proposed. The dolomite based TCES system (Figure 1) can be integrated with a Concentrated Solar Power (CSP) plant to reduce the cost of renewable energy generation and address issues related to the intermittent nature of the solar energy. The same system can be utilized as a Carbon Capture and Storage (CCS) unit to capture the carbon dioxide (CO_2) emissions from the conventional fossil fuels based power plants, and in the process prevent CO_2 from entering the atmosphere.

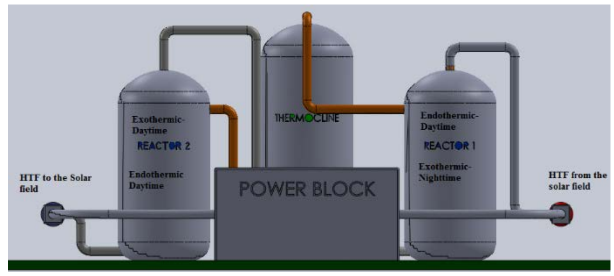


Figure 1. Proposed Thermochemical Energy Storage

This proposal is based on our prior experience in developing thermal energy storage (TES) systems. We propose to test the feasibility of utilizing dolomites, a by-product from phosphate mining, as the energy storage material in a packed bed reactor for the reversible MO/MCO_3 (M = Ca, Mg) reaction. Based on our initial calculation, the $CaO/CaCO_3$ storage system has an energy density of 4806 MJ/m^3, which is within the target range of DOE goal. However, previous attempts to utilize this reaction by other group has not been successful as it suffered from a decrease in capacity over repeated carbonation/calcination cycles [19]. This is mainly due to the sintering of CaO under severe calcination conditions and filling of the pores in CaO due to the formation of the $CaCO_3$ layer which prevents the diffusion of CO_2 to the CaO layers below it.

The use of mined dolomite ore may be advantageous in the present case as the impurities in the raw dolomite ore will increase the surface area for the solid-gas reaction and prevent the sintering of the CaO and MgO particles under harsh calcination conditions. This will allow the TCES system to operate at high pressures and temperatures with no degradation in capacity on repeated thermal cycling, which is not possible with current available technologies. In order to ensure success of this concept, the proposal consists of two independent parts. The first part will focus on studying the decarbonation/carbonation of raw dolomite $MO/CO_2/MCO_3$ (M = Ca and/or Mg) ore with a very high and steady cyclic conversion rate. The basic reactions of the MCO_3/MO system are:

(1) Calcination: $CaCO_3(s) \longrightarrow CaO(s) + CO_2(g)$ $\Delta H° = 178$ KJ/mol (endothermic)
Carbonation: $CaO(s) + CO_2(g) \longrightarrow CaCO_3(s)$ $\Delta H° = -178$ KJ/mol (exothermic)

(2) Calcination: $MgCO_3(s) \longrightarrow MgO(s) + CO_2(g)$ $\Delta H° = 118$ KJ/mol (endothermic)
Carbonation: $MgO(s) + CO_2(g) \longrightarrow CaCO_3(s)$ $\Delta H° = -118$ KJ/mol (exothermic)

It is assumed that the impurities in the mined dolomites has the potential to disrupt the CaO lattice structure resulting in higher reactivity which will allow us to address problem of decrease in capacity over repeated carbonation/calcination cycles faced by other researchers in the past due to sintering. We will determine the enthalpy of the above mentioned carbonation and decarbonation reactions with the dolomite ore and observe the enthalpy variation after repeated cycling.

The second part will be focused on developing a hybrid dolomite system with dopants or supports to introduce defect crystal lattice in the active materials. This is to address extremely slow nature of carbonation reaction and speed up the reaction. We will investigate the use of dopants, such as NaBr and other alkali metal carbonates. LiBr have been recently reported to be very effective in making MgO reactive by a research group at the Tokyo Institute of Technology [20].

6. Past and Present Work Done on Problem by Principal Investigator
Dr. Jaspreet Dhau, faculty mentor of Chemistry is involved in the development of thermal energy storage systems since 2012. He has successfully developed latent heat thermal energy storage systems that can store heat in phase change materials in the temperature range, 300-850 ^{o}C [15, 21-23]. His development has led to a grant of one US patent and filing of three patent application. Dr. Sesha Srinivasan, faculty mentor of Physics has done extensively the thermogravimetric analysis (see Figure 2 below) of ceramic fabric (alumina or yttria) impregnated calcite and dolomite for CO_2 capture (unpublished works [2008-2009] and Graduate Thesis of Drupatie Latchman and Man Su Lee [8, 24, 25]). Dr. Sesha Srinivasan and co-workers have published a research article on hydrogen and carbon dioxide storage via nitrogen rich porous aromatic framework (NPAF), for example, p-phenylenediamine) with the BET surface area of 1790 m^2g^{-1} [26] and at temperatures of 77K and 273K respectively (Figure 3).

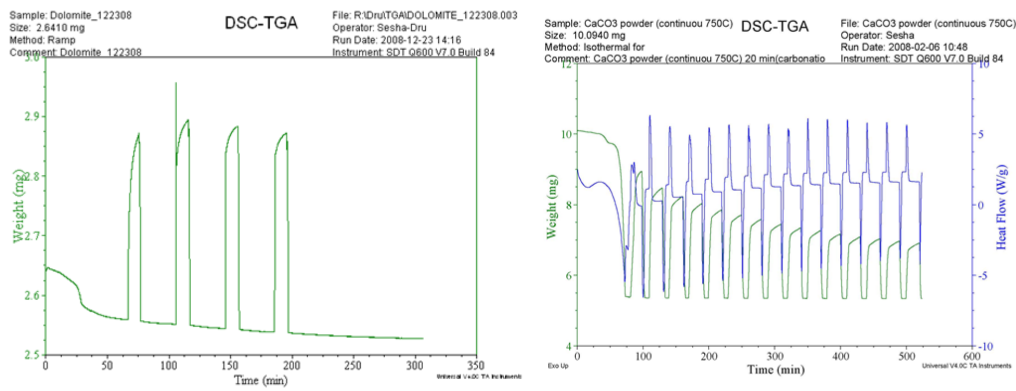


Figure 2: Thermogravimetric analysis of reversible calcination and carbonation cycles of calcite and dolomites at elevated temperatures (750 ^{o}C) [8, 24].

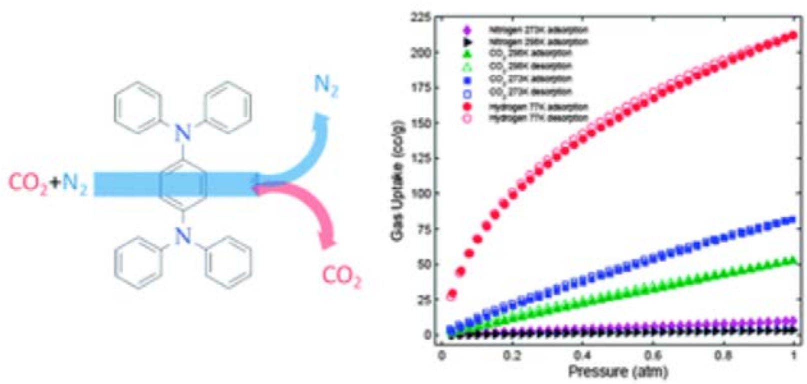


Figure 3: Hydrogen and CO_2 capture at 77K and 273K using nitrogen rich porous aromatic framework [26].

Description of Proposed Research - Methods, Design, and Procedures

The overall objective of this proposal is to develop an inexpensive and efficient sorbent systems not only to capture the greenhouse gas (GHG), CO_2 but also efficiently deployed as thermo-chemical energy storage material in concentrated solar power (CSP) plants. The central hypothesis of the proposed study is to explore the feasibility of waste contaminant in phosphatic rocks such as dolomite (calcium magnesium carbonate) in clean energy applications mentioned above. The following objectives and feasibility studies will be tested.

1) Develop an Inexpensive sorbent materials using waste contaminants from Phosphatic Rocks: Our preliminary investigations revealed that the use of dolomite in CO_2 capture enhances in terms of reaction rate and capacity than pure calcium carbonates since magnesium carbonate with higher surface area, and decomposition at lower temperatures in the dolomite that allows more CO_2 molecules to react with calcium oxide [7].
2) Quantify the CO_2 capture or conversion, determine the regeneration time needed and evaluate the cyclic performance of the dolomite sorbent. CO_2 molecules from the flue gas react calcium oxide in the sorbent to form calcium carbonate. Heat is then used in the regeneration process to drive the CO_2 molecules from the sorbent leaving a pure stream of CO_2 that can be reused or stored. The multicycle conversion can be calculated using the TGA analysis at carbonation/calcination conditions and also fitted with semi-empirical equation [8].
3) Develop a thermochemical energy storage system using the dolomite sorbent material, optimize its characteristics for the yield, reaction rate and high temperature of cyclic processes for the concentrated solar power plants. We will work with our collaborators at University of South Florida, Clean Energy Research Center for utilizing their in-house CSP plant.

The different tasks and research schedule are given in section 6 (Task Breakdown) and 3d.

Plan for Carrying Out Proposed Project

Methodology

The feasibility study to develop dolomites for CO_2 capture and thermochemical energy storage will follow the general methodology used for
(i) Dolomite processing to remove dirt and other contaminants
(ii) Catalyst preparation, for example alumina impregnated dolomite/calcite
(iii) Procedure for carbonation and calcination cycle
(iv) Characterization of dolomites at every stages of calcination and carbonation cycles

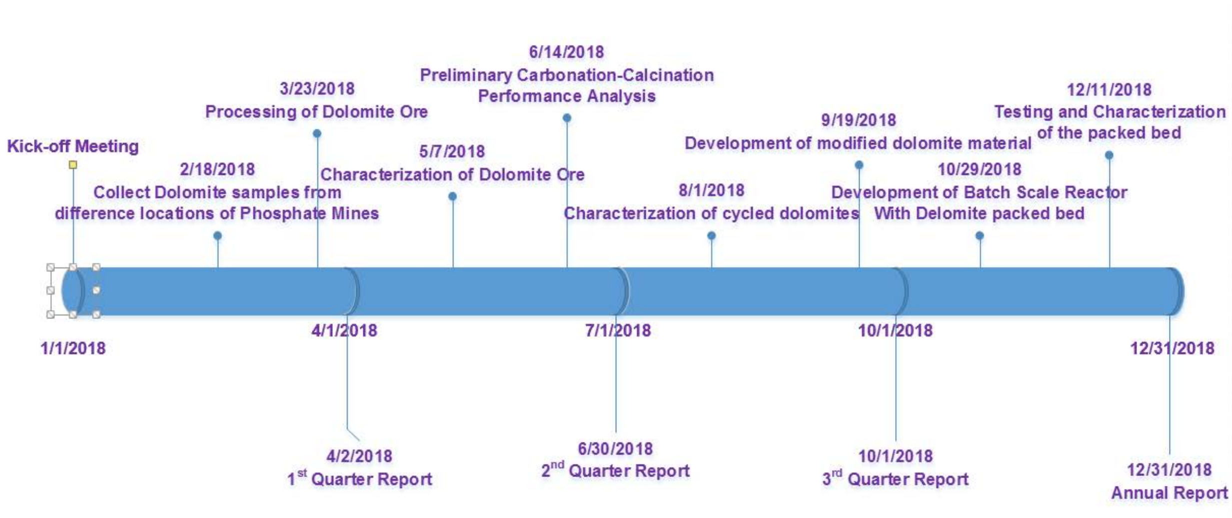
The following high-end tools and equipment will be used to study the characteristics of dolomite materials as an efficient thermochemical energy storage and CO_2 capture applications.
(i) High Energy Ball Milling to pulverize the as-prepared sorbent catalysts
(ii) TGA/DSC Analyses – To estimate and evaluate quantitatively the weight loss and heat flow during calcination and carbonation loop cycles.
(iii) XRD, XRF, FTIR, SEM, BET – To understand the structural, microstructural, chemical, surface area information of dolomites at every cycle or intermittent cycles.

- **Personnel** – SPS members, Florida Polytech Chapter's President Danah Velez and Secretary Wyatt Liptak will be involved in the proposed research and work with the faculty adviser Dr. Sesha Srinivasan. Danah and Wyatt will get proper training on the state-of-the-are equipment and also will get training how to conduct research in a professional way.
- **Expertise** – Danah and Wyatt has necessary background completion of courses like Nanoscale and Instrumentation which are helpful to achieve success in the proposed project.
- **Research space** – A wet chemical research laboratory and a nanotechnology laboratories in Florida Polytech campus will be utilized for the proposed project study. These students also work with our research institute, Florida Phosphate Research for obtaining dolomites for research.

- **SPS Faculty Adviser** Dr. Sesha Srinivasan with his expertise on the proposed project will enable the success of the proposed project from its starts till the end of the project timeline. Dr. Srinivasan also very successfully previously receiving SPS Research Award when he was tenured at Tuskegee University.

Project Timeline

Research Schedule



Tasks Breakdown:

Task 1. Processing of Dolomite ore: We propose to process the dolomite ore to remove dirt and other easily separable contaminants. The clean dolomite will be grinded to obtain a finely and high surface area powdered ore. High surface area reactants facilitates solid-gas phase reaction.

Task 1.1. Process mined dolomite ore to remove dirt and other contaminants.

Task 1.2. Grind the ore, obtained from Task 1.1, by ball milling to obtain a finely and high surface area powdered ore.

Task 2. Characterization. The following techniques will be used for the characterization of the finely powdered ore:

a) XRD to determine the extent of crystallization of the $CaCO_3/MgCO_3$ content in the processed ore.
b) SEM for studying the surface morphology before and after cleaning and grinding.
c) EDX for determining the %age composition of the processed ore.
d) BET surface analyzer for determining the surface area of the processed ore.

Task 3. Preliminary performance analysis of the processed ore in the calcination-carbonation reaction cycle. This will be done by performing TGA experiments under variable reaction conditions which include different pressures and gas flow-rates of air/N_2 and CO_2. Through these TGA experiments the optimum temperature of carbonation and decarbonation of dolomite will be ascertained. The extent of the calcination-carbonation reaction will be determined by observing the weight loss/gain of the reactant materials. The % composition/amount of the active materials ($CaCO_3/MgCO_3$) will be established and the results will be compared with EDX analysis.

Task 4. Characterization. The following techniques will be used for the characterization of the thermal cycled dolomite ore:

a) XRD to determine the extent of crystallization of the $CaCO_3/MgCO_3$ content in the tested ore and compare the result with that from Task 2.
b) SEM for studying the surface morphology before and after thermal cycling.
c) BET surface analyzer for determining the surface area of the ore after thermal testing.

Task 5. Development of modified dolomite material:
The carbonation reaction is thermodynamically favored, however it is extremely slow in nature and the challenge may be to speed up the reaction. In this task, we plan to develop a hybrid dolomite system with dopants or supports that introduces defect crystal lattice in the active materials which would increase chemical reactivity of the mined dolomite and improve the reaction kinetics.

Task 5.1. Develop modified dolomite and investigate its effect on the reactivity of the dolomite ore during carbonation.
Task 5.2. Use alkali metal carbonates as dopants. Subject the mixed metal carbonates to pyrolysis to get molecularly dispersed mixed oxides. The molecularly dispersed mixed alkali metal doped Calcium and magnesium oxides possess high degree of defect crystals providing excellent chemical reactivity.

***Task 6. Costing and Economic Analysis**:* Estimates of the capital and operating expenses for the processing of the mined dolomite ore will be developed. From the size and operating conditions equipment costs will be estimated using the Aspen Process Economic Analyzer module. This module will generate preliminary estimates for the total capital investment and the operational costs to make the dolomite ore suitable as thermochemical/CO_2 sequestration materials.

Budget Justification

The SPS budget we request $2000 to cover the materials and supplies required for the proposed project. We will buy chemicals, solvents, acids, crucibles, TGA pans, glassware and tubes to execute the project.

We have also requested from Florida Industrial and Phosphate Research Institute for additional budget to pay for undergraduate students' salaries and they agreed to support the two students Danah and Waytt if we secure $2000 SPS Chapter Research Award.

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