



The work was submitted to the

Institut für Textiltechnik of RWTH Aachen University

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Production of synthetic Moon soils and their fiberization for lunar settlement applications.

Presented as: Bachelor Thesis

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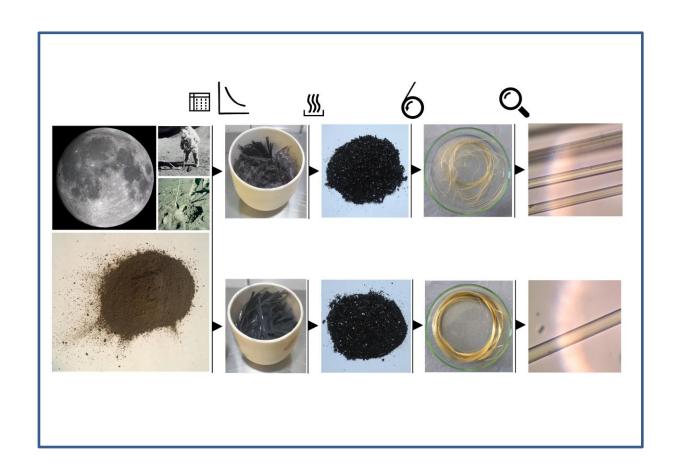
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Aachen, September 2016







2016 BACHELOR THESIS PRODUCTION OF SYNTHETIC MOON SOILS AND THEIR FIBERIZATION FOR LUNAR SETTLEMENT APPLICATIONS ALVARO BLAY SEMPERE

Kurzfassung

Die Vision der Raumfahrtindustrie beinhaltet eine Besiedelung anderer Planeten. Nach Jahrzehnten der exklusiven Roboter-gestützten Raumforschung haben neue Entwicklungen und Rivalitäten nicht nur das Interesse für bemannte Missionen wieder belebt, sondern auch für permanente Stationen auf dem Mond und darüber hinaus erweckt. Diese Siedlungen wären sehr teuer, aufgrund der hohen Kosten für den Transport einer hohen Nutzlast in eine Umlaufbahn und dann zu den Satelliten. Deshalb müssen neue Technologien für eine bessere Wirtschaftlichkeit geschaffen und verbessert werden.

In diesem Projekt wird die Machbarkeit der Herstellung von Basaltfasern aus Mondgestein untersucht. Fasern zeigen eine große Vielseitigkeit bezogen auf ihre Anwendungen, da sie im Gegensatz zu Monolithen eine gewisse Flexibilität aufweisen. Sie können u. a. als Verstärkungsbaumaterial verwendet werden. Daher wird in dieser Arbeit untersucht, inwiefern eine Faserherstellung aus Mondsand möglich ist. Daher wurde die Zusammensetzung bestimmter Mondgesteine neu erstellt, und ein Spinnfenster bestimmt.

Schlagwörter: ISRU, Simulant, Fasern, Mond, Basalt.

English Abstract

Space industry is about to take another direction. After decades of exclusive robotic space exploration, new developments and rivalries have revived the interest for manned missions and permanent bases on the Moon and beyond. These settlements would be very pricey, due to the high cost of bringing a payload into orbit and then to the satellite. Therefore, new technologies and applications must be created and improved for a better cost-effectiveness.

The aim of this work is to determine the feasibility of producing basalt fibres from lunar sand. Fibres show a large versatility on their applications, as they may be processed at will. They can be used as construction material reinforcement, or as fabric, furniture, chassis, etc. For that reason, checking if it is possible to use lunar basalt for fiberization is an important step for *in situ* Moon resource utilization. Firstly, the composition of certain Moon sand was recreated and the expected fiberizing window was studied in order to produce fibers by spinning.

Keywords: ISRU, simulant, fibers, moon, basalt

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Abbreviations

ISRU In situ resource utilization

LEO Low Earth Orbit

GCR Galactic Cosmic Radiation

SPE Solar Proton Event

XRF X-ray fluorescence

INAA Instrumental Neutron Activation Analysis

RNAA Radiochemical Neutron Activation Analysis

APXS Alpha Particle X-ray Spectrometer

JSC-1 Johnson Space Center 1

CAS-1 Chinese Academy of Sciences 1

FJS-1 Fuji Japanese Simulant 1

MLS-1 Minnesota Lunar Simulant 1

Basfiber® Basalt Fiber from Kamenny Vek

ESA European Space Agency

ISS International Space Station

MT Metric ton

ITALUS-1 ITA Lunar Simulant 1

ITALUS-2 ITA Lunar Simulant 2

1 Introduction and Objectives

Bringing a spacecraft into orbit constitutes a tremendous share of the total costs of every space mission. For this task to be done, humanity has relied on Rocket Science ever since the first satellite was placed into orbit, while other alternative disciplines have not been able to become real or fulfil the requirements, such as the concept of space elevator. Rockets are the only used means of transportation into space, and even though they are subjected to technological improvement and optimization of costs, they are not expected to be replaced by other technology anytime soon. The next decades of space exploration will be strongly bounded to the restrictions of rockets and humans must learn how to adapt to these limitations: the high costs of launching and the impossibility to launch very heavy loads.

Current costs of launch to Low Earth Orbit (LEO) range from the 2719 USD/kg for SpaceX's Falcon 9 to the 8730 USD/kg for Ariane 5ME, bringing a maximum payload of 22800 kg and 25200 kg respectively. The maximum allowed payload per vehicle for a further away body, like Mars, is notoriously smaller, 4500 kg. The last generation of rockets is very efficient compared to their predecessors, but the mass limitation is still challenging, and this is an obstacle in the direction the space industry is taking. [www12a] [www16a]

In the last years some relevant countries and enterprises have joined the traditional space agencies in the access to space and have changed the scenario: manned missions beyond LEO are now a part of the discussion. On one side, China sent probes to the Moon not long ago and has expressed its interest in manned missions to the Moon and Mars. On the other hand, the American company SpaceX has revolutionised the industry as a successful private rocket and capsule manufacturer. ESA is, among other projects, working on a future "Moon village" (Figure 1.1), which could become the first continuous human settlement on our satellite. [www13a] [www16b]

The previous weight restrictions are a problem for these interests: manned space-craft needs considerably larger space and equipment for life support on board than the unmanned ones, and this is translated in more weight to launch. Humans in an exploration mission on another planet or moon would need to bring everything they need with them, especially if they intended to settle down. Lightweight design has been a key discipline in the solution for these issues, but it is not always enough. That is why *in situ* resource utilisation (ISRU), the study of how materials in those places can be used, has got relevant and it has been specially promoted by space agencies, especially NASA. [www15a]

This project will study a certain way of ISRU: the usage of Lunar soil for the production of fibres. Fibres are an excellent material that can be useful in these conditions, as they are very versatile and they can be shaped easily at will. Taking advantage of the inorganic resources of the Moon this way could help reduce the overall costs of

transport, since once the required machines were brought there, a large amount of fibres would be able to be produced.

It is not possible to use actual Lunar sand for the experimentation of ISRU here on Earth, and the simulants are costly, depending on the application, therefore this thesis will also test a method to produce a composition-wise synthetic lunar soil from commercial basalt and selected additives. A composition-wise simulant would emulate the moon regolith in terms of the chemical composition and it could suitable for certain tests, even though other properties of this material are not the same as those of the Moon, like the grain size or crystal structure.



Figure 1.1: ESA's Moon village concept. [Dav15]

2 Literature Search

In situ resource utilization (ISRU) is a discipline in which many experts have been working for decades. The use of extraterrestrial materials in an optimal, original and alternative way is attractive for many scientists and engineers, whose aim is to push the limits of knowledge, and an extensive and imaginative literature can be found after some research.

The most substantial objective of ISRU is to reduce the costs of launching for future missions. The present costs of launch to Low Earth Orbit (LEO) for some of the most advanced launch systems vary from the 2719 USD/kg for SpaceX's Falcon 9 to the 8730 USD/kg, and they get even higher for launches to the Moon or other planets. [www12a] [www16a] Profiting from the resources of planets would be extremely useful for modern and future missions, and having lower-cost missions would increase the number of them.

The study of ISRU imposes no limit for the resources that can be used or not. The first kind of resource that comes to mind is the use of rocky solids, like the regolith. However, it covers much more. ISRU is not a synonym for the use of moon rocks, or Martian soil, but also means to take advantage of the diverse matter in different physical states that can be found. Several projects deal with the extraction and handling of stony solid resources [ZMM08] [BBE05], but many others try to approach the problem in other unconventional ways: using water ice from the Martian poles to build igloo-like habitats [CML16]; extracting water ice from the Moon [www09a]; extraction of atmospheric resources, like converting CO₂ from the Venusian and Martian atmospheres into oxygen [MS03]; extracting liquid ethane/methane from the lakes in Titan, etc.

The projects focused on the use of rocky resources, like the present thesis, often need to rely on the usage of soil simulants. Simulants are man-made or man-processed terrestrial soils that keep a resemblance with the real extraterrestrial soil in some ways. With simulants, one may be able to perform several tests that, otherwise, would be impossible to do: real samples from the Moon or other planets are scarce, unavailable for third parties or simply inexistent. For instance, during the years of service of the Apollo missions, NASA was only able to bring 382 kg of material to Earth, [www15b] and there are no samples of the Martian surface on Earth, apart from some meteorites that were formed in the planet and then ejected by the impact of an asteroid. [www16j]

Lunar simulants are less rare than real samples, but their offer is also low. The NASA-designated company ORBITEC has produced the lunar simulant JSC-1 for years, but they finished their stock and never replaced it. However, they still sold Martian soil simulant until at least 2014. [www14a] Other private companies, universities and space agencies have taken over and decided to produce their own simulant for their own purposes. [MCB94] [SCS05] [ZWL05]

The first part of the project will be focused on the obtainment of a lunar simulant, which is required for our own experiments. Simulants are usually extracted from coarse volcanic rock and then they are mechanically milled in order to get the desired grain size. [SCS05] In this project the artificial soil will be produced by melting commercial basalt together with some additives, which will modify the chemical composition of the initial basalt into the Moon composition.

Table 2.1: Composition of different moon samples by the Apollo missions and a basalt sample from Earth

	Sample number and type of material									
	14163 (basaltic soil)	78221 (basaltic soil)	10084 (basaltic soil)	67215 (polymict breccia)	Basfiber® (terrestrial basalt)					
SiO ₂	47,30%	43,67%	42,16%	0,00%	55,69%					
Fe ₂ O ₃	0,00%	0,00%	0,00%	0,00%	10,80%					
Al2O3	17,80%	17,13%	13,60%	24,30%	15,44%					
MgO	9,60%	10,55%	7,76%	6,80%	4,06%					
CaO	11,40%	11,79%	11,94%	14,90%	7,43%					
Na₂O	0,70%	0,37%	0,47%	0,30%	2,40%					
Cr ₂ O ₃	0,20%	0,00%	0,00%	0,00%	0,00%					
TiO ₂	1,60%	3,84%	7,75%	0,43%	1,23%					
K ₂ O	0,60%	0,09%	0,16%	0,00%	1,51%					
FeO	10,50%	11,68%	15,34%	7,95%	0,00%					
MnO	0,10%	0,16%	0,20%	0,00%	0,00%					
P_2O_5	n.d.	0,08%	0,05%	0,00%	0,00%					

In the Table 2.1 the composition of different samples taken by the Apollo is shown and compared to the composition of a terrestrial basalt. Due to the fact that one of the major changes in the composition will be an increase of iron oxides to roughly 15% of the overall composition, a project related to the effect of iron oxide content on the crystallization of diopside glass-ceramic glaze was of greatly useful. [RRA02] The main concept of this literature work consisted on mixing iron-less diopside frit with granite, which has a higher ferric oxide content (17,79%wt), in different proportions (from 10%-90% to 100%-0%), and see how this affected the final structure. The positive results of this work backs up the method of mixing and melting and sets a starting point for the project.

Other relevant literature to this project is previous trials on characterization, glass formation and selected glass properties for lunar simulants. The amount of research in this area is not extremely extensive, but the successful research by Ray *et al* [RRS10] showed that it is possible to proceed with the formation of fibers out of lunar simulants, as well as hollow glass microspheres, as shown in Figure 2.1.

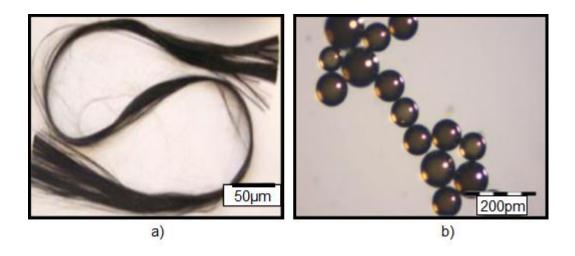


Figure 2.1: Glass fibers (a) and hollow microspheres (b) produced from JSC-1 lunar simulant. [RRS10]

The differences in composition and crystal structure between lunar soil and simulants are considered to be major in some aspects, and therefore the results have to be analyzed carefully. Simulants, as they suggest by their name, are supposed to simulate the lunar soil in some of its characteristics, not to copy them all perfectly. The only procedure which would yield no error would involve using real samples from the lunar surface, or produce a perfectly accurate simulant. The latter is a difficult accomplishment, as ferrous oxide (FeO), the only oxidation state of iron found on the Moon, is a very rare iron oxide on Earth, where ferric oxide (Fe₂O₃) is the common one. The lack of a highly oxygenized atmosphere on the Moon allows ferrous oxide to exist, but in our planet, Fe is usually found in its fully oxidized state. [TL10] In other words, lunar samples would change their composition under the influence of an oxygen rich atmosphere like Earth's or a theoretical lunar habitat, as their Iron (II) oxide would most likely transform into Iron (III) oxide to some extent or completely. This oxidation process would bring the moon material to a composition in which there is a greater experience, that is, with a high content of Fe₂O₃, like simulants or other basaltic rocks on Earth. This process, would however consume considerable amounts of oxygen, presumably, and it would be challenging for early and small human settlements on the Moon with limited resources. Obtaining oxygen from the lunar water ice reserves could solve the problem, but the insights of extraction of breathable and functional oxygen from the Moon is not in the research boundary of this project.

The possibility of producing fibers with generic lunar soil is a reality, given the current data and research mentioned, and this discipline has the potential to play a major role in the ISRU of the satellite. One of the most studied and useful fields of study in ISRU involves the 3D printing techniques, [www14b] which can be complemented with the Textile Engineering in many aspects. The study of 3D printing applied to the Moon has yield projects about habitation and infrastructure, radiation protection covering, surface paving, bridges, dust-shield walls and landing fields, among others. [LAR15]

Textile engineering, on the other hand, has other applications, such as reinforcement of concrete structures [SPM05], vacuum infusion processes, clothing, insulation, etc. Materials created with the help of textile engineering would be more suitable in many aspects to those produced with 3D printing. This would translate into a variety of techniques to be used on the endemic material, which would lead to a more efficient exploitation of resources.

The spinning process for the production of fibers can be feasible on the Moon with the use of electrical heaters and the correct materials. The first step on any fiber production process starts with the analysis of the chemical composition and properties of the precursor material, as it determines the properties and applications of the fibers. [PSG10] Different types of glass fibers can be made by altering the composition of the starting glass, which basically consists of many different oxides. Different types of glass, like E glass, A glass, ECR glass, etc. can be produced when each quantity of the oxides of their composition lies between certain values. Further differences in the composition may lead to other kind of fibers, such as basalt fibers, with totally different properties and applications. Once a composition is selected and grinded, it undergoes a series of processes. The first one, the melting process, helps to the homogenization of the mixture and is fundamental to obtain a constant fiber quality. It is possible to execute this right before the spinning of fibers takes place (direct melt process), in the same machine, but it may be done independently of the spinning process as well, producing marbles or frits which can be later melted again and fiberized in the spinning machine (marble melting process, Figure 2.2). Both processes are common in the industry and its selection depends on the user's needs. The latter is more useful in the case of this project, as the initial melting would produce frits that could be studied and worked with, serving as a lunar simulant, and not directly transformed into fibers.

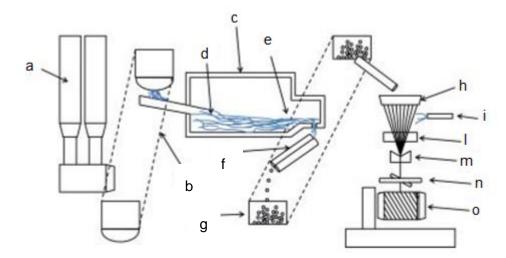


Figure 2.2: Marble melting. a) Mixing silos; b) Furnace; d) Melt; e) Refiner; f) Marble forming; g) Transport; h) Marble bushing; i) Water spray; l) Sizing applicator; m) Strand formation; n) Transversing; o) Cake winder. [PSG10] [Hea01]

The fiber forming itself consists on drawing off fiber from a crucible, which is a Platinum-Rhodium piece that allows the flow of melted glass in the direction of gravity, attenuating the filaments while doing so. In order for a fiber to be fiberized, the viscosity has to range from 30 to 100 Pas, and the required temperature to get such a viscosity shall be computed before the fiber forming process. Once this temperature range is reached, the bushing takes place, in which the compensation of gravity, friction and fluid mechanic terms allows the melted glass to go through a small hole, which gives the fiber an initial diameter. This diameter can be later sized into a smaller radius with the use of a rotating applicator, which draws the fiber off the crucible as it spins. The resulting fiber is winded in a strand and may be post-processed afterwards.

The mechanical properties of the produced glass or basalt fibers can be determined given their diameter of cross section, tensile strength, elastic modulus and elongation at break.

3 Economic impact of in situ resource utilization in a Moon base

Projects for a Moon base in the near future are being planned and discussed by several space agencies and space companies from different nations. The European Space Agency (ESA) has plans for this project to be accomplished in the 2030's decade, [Dav15] a very early date in the long time plans people are used to in the space industry. A group of space experts, entrepreneurs and venture capitalists participated in 2014 in a meeting at Draper-Fischer-Jurvetson, hosted by Steve Jurvetson himself. [HNP16] The purpose of this meeting was to analyze and determine an economically feasible strategy for the settlement of 100 people in the Moon within 15 years, whose first step would be to build a core to support 10 people within 5 years. A limitation on the cost was also set: 5 billion USD. The early discussions already showed that life support would be critical for the success and affordability of the enterprise. The main factors for life support among many interacting variables were determined:

- Number of people
- Duration of stay
- Availability of indigenous supporting resources (ISRU)
- Readiness of the physical-chemical and bioregenerative technologies
- Ease and cost of resupply from Earth, provisions for emergencies.

As it can be noticed, IRSU is one of the most critical strategies for this ambitious programs, and also one of the disciplines with the most diverse applications in the Moon settlement plans: for the acquisition of solar energy, Lunar-based self-replicating solar factories using lunar materials can be designed; [Lew16] for the obtainment of metals, the processing of regolith in the search for metallic micrometeorites present [Win16], etc.

What can also be spotted from these projects is the clear division in the two phases. A lunar settlement with an initial crew of 100 people is hardly feasible, technologically and economically. These projects aim for the accomplishment of certain initial goals for a lunar habitat that would later be expanded.

Lunar Station Initial Goals								
Pressurized volume	900+ m3							
Habitable volume	300+ m3							
Power	100+ kW							
Initial crew size	6-10 people							
Life support recovery	90% or better							
	Every 6							
Crew rotation	months							
Initial landed mass	150+ MT							
Initial yearly resupply	30+ MT							

Table 3.1: Lunar Station Initial Goals. [PHN16]

In the Table 3.1 the initial goals of the Lunar station are shown. The initial landed mass of the habitat of 150 MT is low in comparison with the International Space Station's (ISS) 420 MT. With the use of prefabricated Bigelow Aerospace habitat modules and using SpaceX's new Falcon rockets, the initial phase of the Moon base would be feasible in 5 years from the day mission were accepted. (Figure 3.1)

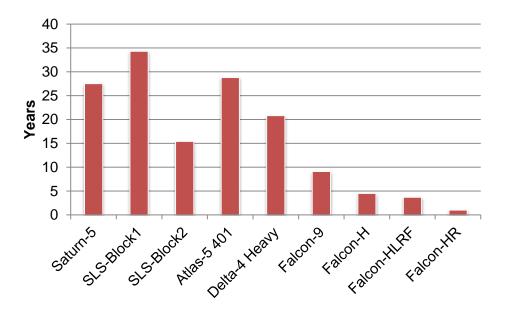


Figure 3.1: Number of years to get 150 MT of equipment and supplies to the lunar surface to begin operating the Lunar Station. [PHN16]

In the first phase of a lunar settlement program, there would however be little room for ISRU. The crew would be focused in bringing habitats together, investigations for resources and hazards, possibilities for food growth, studying the potential of the chosen site for industrialization, mining and further expansion, medical sciences testing, etc. [Win16] [PHN16] The real impact of ISRU would take place in the second phase of

the project, when a larger population will require a higher independence from Earth supplies.

One of the first tasks of ISRU will consist on the covering and stabilization of lunar soil over the existent or new modules, in order to protect the crew from outer radiation and serve as a preliminary thermal insulator. The estimation of required material for this duty is the first indicator of the potential savings ISRU can provide.

To estimate the amount of material needed for this coating, it can be assumed that the initial base would consist of cylindrical habitats connected to each other, with a total inner volume of 300 cubic meters. With a speculated 2,5 m in diameter for the cylinders and assuming they are laid in a straight line, the habitat would be 61,12 m long in total. According to Miller *et al*, a covering of 46 cm would afford substantial protection against primary Galactic Cosmic Rays (GCR) nuclei and Solar Proton Event (SPE) protons. [MTZ09] Galactic Cosmic Rays are stable charged particles, usually H⁺, that have been accelerated somewhere in the universe and reach our planetary neighborhood. Their energy lies between 10⁹ eV and 10²⁰ eV, depending on their source (black holes, galactic nuclei...). Solar Proton Events are caused due to solar flares, which accelerate protons and other ions and eject the particles in many directions. And often involve energies over 10⁹ eV.

Using the 46 cm covering, only reduced residual dose was measured, originated from the surviving charged fragments of the heavy beams. [MTZ09] For this estimation, therefore, the whole module cylinder will be considered coated with a 46 cm wide layer of lunar soil. (Figure 3.2)



Figure 3.2: Model used for the calculation of regolith volume needed for radiation shielding.

The calculated total volume of the coating would be 120,31 m³, that is, with an average density of 1.71 g/cm³, about 206 tons. In order to put this number into perspective, in the Table 3.2 it is computed the number of rockets, costs and time required for sending additional 120 metric tons to the Moon. It would require at least 29 Falcon-HR (rockets still in development) and cost additional 248 million USD to do so, and this would be just for the first phase of the lunar base. The launch from Earth of 120 tons just for radiation shielding is not feasible.

Table 3.2: Number of rockets, costs and years required to send radiation shielding for the initial base.

	Rocket type										
	Saturn-5	SLS-Block 1	SLA-Block 2	Atlas-5 401	Delta-4 Heavy	Falcon-9	Falcon-H	Falcon HLRF	Falcon-HR		
Cost per lunar launch (M\$)	3100	2125	1817	246	525	92	188	358	31		
Payload to Moon(MT)	16,40	8,80	16,30	1,20	3,60	1,50	6,00	13,50	4,20		
Launches/year to Moon	0,33	0,5	0,6	4,25	2	11	5,5	3	35		
Launches to get 120 MT to the Moon	8,0	14,0	8,0	100,0	34,0	80,0	20,0	9,0	29,0		
Cost to get 120 MT to the Moon (M\$)	24800	17000	14536	1968	4200	736	1504	2864	248		
Years to get 120 MT to the Moon	24,2	28,0	13,3	23,5	17,0	7,3	3,6	3,0	0,8		

It should be mentioned, however, that the radiation shielding, in case it was brought from Earth, would be made out of another material, even water. That would decrease the costs mentioned before, but they would still be an economic disadvantage, as fairly available and virtually free material which suitable for this assignment is present on the Moon already.

The initial savings of this initial ISRU application, which would be at least of 736 million USD for Falcon-9, the only one of the Falcon rockets already manufactured, could mean the difference between an approved mission or a cancelled one. If further expansion of the habitat took place, and a permanent "moon village" established, these savings would be not only crucial, but possibly obligatory.

It is hard to estimate the economic impact that other ISRU applications would have, like production of fibers. That would depend enormously from the design of the settlement habitat, the base crew needs, their required equipment, etc. However, the same principle as the radiation shielding applies: bringing materials from Earth for certain duty that can be successfully done with lunar materials translates into an inevitably loss of money. Fibers could serve as an excellent material on a lunar settlement, as it will be discussed in the following chapters, and they would, in any case, save money and give access to the manufacturing of many other kind of materials of great use.

4 Composition of the lunar surface

Simulants of different materials from space have been developed since the beginning of the space exploration in order to study the behavior of materials whose presence on Earth is extremely limited. In absence of useful quantities of lunar soil for experiments, artificial simulants must be produced.

The synthesis of artificial Moon sand can be done in several manners, depending on the objective of the product. For instance, a certain study such as the design of the wheels of a rover would set as a priority to emulate the same grain size, but not the composition; or maybe to emulate the pigmentation of the sand for visual recreation, where the chemical composition or magnetic properties are irrelevant. In this project, the emulation of the soil is composition-wise, as it is, among others, the most important parameter for the production of fibers. Other characteristics, like the crystallography, are not taken into account, and this is a source of error in some cases: simulants do not usually emulate the lunar soil completely.

In Table 4.1, in the next page, it is shown the partial results of a survey on the relative importance of different needs for the lunar soil simulant for 35 scientists involved in projects where simulants are required. [Sim10] The characteristics that most of them value are the physical characteristics, size, shape, particle size and grain size, with over 70 % of projects requiring these resemblances. This is because many of these projects are related to the extraction and transport of material for mining. Composition similarity is not a priority for over 50 % of the projects, with only 45,71 % of projects needing it in their experiments.

The determination of the composition of the Moon surface is a key factor to this project, as it is the major need in our case. This chapter will evaluate the validity of the analyses of Lunar samples in existence, in order to check the reliability of this data based on the instruments that were used for its determination. It will also cover the selection of certain Moon soil composition, the reasons behind the decision, and the process to manufacture this soil simulant out of commercial basalt fibers and other additives.

Planets and other celestial bodies are not homogeneous, and the Moon is not an exception. The lunar samples subjected to a chemical composition analysis represent the composition of small points in the surface, and it is possible to assume that the chemical composition varies along the surface of the Moon, as well as it does on the Earth.

The Moon is nowhere near homogeneous in its composition: many samples have been taken from different regions by several space missions and they all show differences in their composition. Most of them coincide in a significant amount of silicates and a similar composition to the volcanic materials that can be found on Earth, but there are considerable differences in the composition that will have to be taken into account in this project.

Table 4.1: Lunar Regolith Simulant User Needs Survey Report. Only characteristics important for more than 40 % of the projects are shown. The composition is important for only 45.71 % of the user's projects.[Sim10]

Lunar Regolith Simulant Development & Characterization Project NASA / MSFC / VP33								
Title: Lunar Regolith Simulant Users Needs	Document No.:	Sumulant-Doc-0	11	Draft Baseline Draft Date 09/01/2008				
Survey Report	Effective Date: T	BD		Page 17 of	17			
Survey Results - Cl	naracteristics							
Total Respondents		35						
Total Characteristics		56						
Total Needs		676						
		Number of	P	ercent of	Total Per-			
		Respondents	Res	spondents	cent of			
		Who Said	V	Vho Said	"Yes" Re-			
Respondents Who Fe	lt That	"Yes"		"Yes"	sponses			
Physical Characteristics	s are Important	32	Ç	91,43 %	4,73 %			
Size is Important		29	·	82,86 %	4,29 %			
Shape is Important		29	•	82,86 %	4,29 %			
Particle Size is Importa	nt	27	-	77,14 %	3,99 %			
Grain Size Distribution	is Important	25	•	71,43 %	3,70 %			
Electrostatic Characteri	stics are							
Important		21	(60,00 %	3,11 %			
Abrasion is Important		21	(60,00 %	3,11 %			
Particle Shape Distribut	tion is Important	21	(60,00 %	3,11 %			
Mineral/Chemical Char	acteristics are							
Important		19		54,29 %	2,81 %			
Magnetic Characteristic		19		54,29 %	2,81 %			
Non-Visible Particles ar	e of Concern	19		54,29 %	2,81 %			
Electrostatic Charging i		18		51,43 %	2,66 %			
Composition is Importa		16		45,71 %	2,37 %			
Conductivity is Importar		16 45,71 %			2,37 %			
Magnetic Grain Propert	ies are Im-							
portant		16 45,71 %			2,37 %			
Thermal Characteristics	15		42,86 % 42.86 %	2,22 %				
"Smoke"-sized Particles	15		2,22 %					
Bulk Density is Importa	15 42,86 %			2,22 %				
Hardness is Important	15 42,86 %			2,22 % 2,22 %				
Thermal Properties are	15	15 42,86 %						
Particle Density is Impo	14	4	40,00 %	2,07 %				
Density is Important	14	4	2,07 %					
Glass Composition is Ir	nportant	14	4	2,07 %				
Hardness is important		14	4	40,00 %	2,07 %			

4.1 Reliability of soil composition data

The Apollo missions gathered and brought to Earth 2200 different samples between 1969 and 1972. [www15b] These samples, in contrast with the samples of other planets, were brought to Earth, where they were able to be studied in depth by many different methods. The vicinity of the Moon permitted the return of 382 kg of material in total, something that has not been done with Mars. Other than meteorites coming from Mars, there is a lack of samples directly taken from the planet, and the analyses have to be performed on the Martian surface, thus reducing the number of tests and methods available, and that requires an appropriate selection of the measuring instruments and spectroscopy adapted to the goals of the mission.

As no specific spectrometer was needed on the Moon for *in situ* analysis, engineers have been able to study the samples with many kinds of spectrometers on Earth and therefore, it has been possible to determine the composition of multiple sand samples with many different methods, thus giving us a clear vision of the composition of this soil.

One of the thousands reference samples from the Moon that have been examined, and the one that will be the subject of study in this project, is called "Lunar sample 10084". It is found to be a mix of around 66% basalt, 5% red brown glass, 22% anorthosite, 8% KREEP and 1% meteorite. Its chemical composition has been determined by different methods, like XRF, IDMSA, INAA, RNAA and wet. Out of these methods, INAA (Instrumental Neutron Activation Analysis) [Eby16], Mössbauer spectroscopy [SKM08] and wet methods [FP85] are effective at determining the content and nature of iron in the soil samples in comparison with the other of methods. The determination of which kind of iron oxides are present is a key factor in this project, because it is one of the biggest differences between terrestrial and lunar basalt compositions.

The oxidation state of iron and oxygen fugacity control is an important part of the characterization of moon rocks and soil. Lunar materials equilibrated at the more reducing Fe-FeO buffer, whereas terrestrial igneous rocks have equilibrated at a higher oxidizing buffer, due to the high amount of oxygen in our atmosphere in comparison with the absence of atmosphere in the Moon. High temperature experiments using lunar simulant materials whose function is to duplicate the conditions on the lunar surface require experimental control of vacuum and oxygen fugacity to the appropriate values and the definitive determination of the oxidation state of Fe is done by the Mössbauer spectroscopy. [BS05]

In the following Table 4.2 the discrepancies about the composition of lunar sample 10084 (Figure 4.1) when using different methods are shown, along with a legend.

Reference weight	LSPET69		Wiesmann76 Gast70		Laul80 bulk		Rhodes81		Agrell70		
SiO ₂	43	(b)				41,3	(e)	41,9	(d)	42,16	(f)
TiO ₂	7	(b)				7,5	(e)	7,56	(d)	7,75	(f)
Al_2O_3	13	(b)				13,7	(e)	13,55	(d)	13,6	(f)
FeO	16	(b)				15,8	(e)	15,94	(d)	15,34	(f)
MnO	0,23	(b)				0,213	(e)	0,21	(d)	0,2	(f)
MgO	8	(b)				8	(e)	7,82	(d)	7,76	(f)
CaO	12	(b)				12,5	(e)	12,08	(d)	11,94	(f)
Na ₂ O	0,54	(b)	0,46			0,41	(e)	0,4	(d)	0,47	(f)
K2O	0,12	(b)	0,145	0,138	(a)	0,14	(e)	0,13	(d)	0,16	(f)
P ₂ O ₅								0,11	(d)	0,05	(f)
S %										0,12	(f)

Table 4.2: Chemical composition of 10084 using different methods. [Mey09]

Techniques:

(a) IDMS, (b) emiss. Spec., (c) radiation counting, (d) XRF, (e) INAA, (f) wet



Figure 4.1: Photography of the lunar sample 10084. [www16h]

As it can be observed, ferric oxide (Fe_2O_3) does not appear in the analysis. Other publications [RRS10] agree on the matter, and affirm that Lunar soil contains only ferrous oxide (FeO). The data related to glass manufacturing processes that include both Iron (III) oxide and Iron (II) oxide are quite limited. [www16e]

The assumptions made in order to overcome this will be explained in the following chapters.

4.2 Selection of the target Moon soil composition

The purpose of this thesis is to study the feasibility of producing fibers out of Moon soil samples, but given the variety of materials that can be found in different regions of the satellite and due to the fact that there is still not proper data or samples for many of these materials, the only feasible way is to work with them with theoretical computation in the ISRU study. It is necessary to restrict the options and work with a suitable composition whose data is reliable and available.

Two possibilities arise from this consideration. One is to select an average composition of the Moon soil and the other is to select a certain Moon sample composition. Choosing an average composition is logical, as the results would approximately match many different compositions all over the satellite, but it is on the other hand inconsistent, as the exact average composition does not exist, and the method would be valid for none of them in particular. The second option is centered in existent data for a real sample, even though it would work worse for others than the average composition option. However, this concern can be minimized by picking a sample that is valid for a large region: this way, the results would be accurately valid for the soil of a large area. These areas exist on the Moon, and the biggest and most known examples are the Mare. These regions are, in summary, solidified seas of lava. They cover an extensive part of the Moon and were created long ago by active volcanism, which flooded large parts of the surface. Because they are all facing to Earth, making direct communication easy, and because they have been the chosen landing sites of the Apollo missions, it is reasonable to deduce that the region will be the scenario of future colonies or bases. [Lin76]

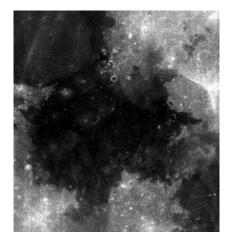


Figure 4.2: Mare Tranquillitatis (the dark area in the center of the picture) is a relatively homogeneous region on the Moon. [www16k]

For the procedures of this project, a well-defined lunar sample has been chosen.the second option has been chosen. The selected composition is that of the Lunar sample

10084, a general soil gathered by the Apollo 11 mission in Mare Tranquillitatis. (Figure 4.2) [www16c] The composition by weight of this sample is shown in the Table 4.3:

Table 4.3: Composition by weight of Lunar sample 10084. [Mey09]

	%wt
SiO ₂	42,16 %
Fe ₂ O ₃	0,00 %
Al ₂ O ₃	13,60 %
MgO	7,76 %
CaO	11,94 %
Na ₂ O	0,47 %
Cr2O ₃	0,00 %
TiO ₂	7,75 %
K20	0,16 %
FeO	15,34 %
MnO	0,20 %
P ₂ O ₅	0,05 %

The determination of the composition was performed using different methods of spectroscopy, of which the wet method Agrell70 was selected, which is shown in Table 4.2, due to the highest accuracy of iron oxides differentiation in comparison with XRF methods.

For the study of what method is best to determine the chemical and crystalline structure of lunar samples, the example of different Martian probes is useful. Because Mars has high amounts of both ferric and ferrous oxide on most of the surface, missions have tried to send the best equipment to determine the proportion of each oxide. Most of the robots sent to Mars have on-board instruments for the characterization of the composition of rocks and sand. The Mars Science Laboratory Curiosity, the latest and best equipped rover ever sent to the planet, has an X-Ray Diffraction instrument (CheMin) and an Alpha-Particle X-Ray Spectrometer (APXS - SAM) [www16f] to determine the amounts of the crystalline and amorphous components of the sand. The XRD analysis is able to give the crystalline structure of the sand, but not the composition by weight. It has to be complemented with the data coming from APXS, due to the fact that APXS is not able to distinguish between oxidation states. When using both instruments, a better vision of the composition of the samples can be obtained, but it is still not possible to determine the exact percentage for FeO and Fe₂O₃. [Gro13] This problematic is shown in Table 4.4, where the content of FeO and Fe₂O₃ is given combined:

Table 4.4: Chemical composition of a Martian sample.

	Origi	n (instrument)	Comp	osition
	APXS	APS + CheMin	Amporphous	Crystalline
SiO2 wt%	42,88	42,88	37,2	47,59
TiO2	1,19	1,19	2,06	0,47
Al2O3	9,43	9,43	6,04	12,24
Cr2O3	0,49	0,49	1,09	0
FeO+ Fe ₂ O ₃	19,19	10,43	23,14	-0,1
FeO-Cryst	-	7,37	-0,01	13,48
Fe ₂ O ₃ -Cryst	-	1,39	-0,01	2,55
MnO	0,41	0,41	0,91	0
MgO	8,69	8,69	4,86	11,86
CaO	7,28	7,28	5,61	8,67
Na2O	2,72	2,72	3,56	2,03
K2O	0,49	0,49	0,89	0,16
P2O5	0,94	0,94	2,09	-0,01
SO3	5,45	4,96	11,01	-0,05
SO3-Cryst	-	0,49	-0,01	0,9
CI	0,61	0,61	1,35	-0,01
Sum	99,77	99,77	99,77	99,77
Σ(FeO+				
Fe ₂ O ₃)	19,19	19,19	23,14	16,03
Σ(SO3)	SO3) 5,54 5,54			0,9
Relative	to whol	45,3	54,7	
Relative t	o XRD	crystalline	-	100

The instruments on-board the Spirit and Opportunity rovers, however, had a Mössbauer spectrometer with them, apart from an APXS. This device uses Gamma rays instead of X-Rays, and it is able to determine iron structures and content. (Figure 4.3) [MMD10]

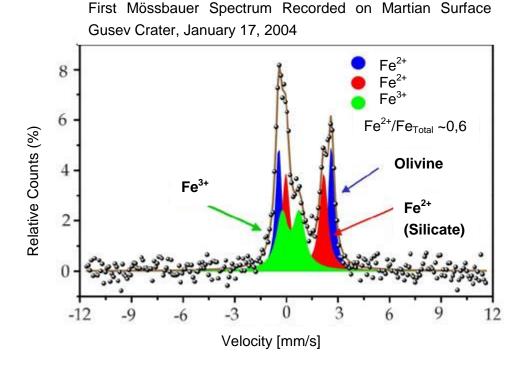


Figure 4.3: Mössbauer spectrum results graph for a Martian sample. [www04a]

Related to the Moon samples, the use of wet methods provide also an understanding on the iron content in its different oxidation states, and that is why Agrell70 is used above the other options. The 10084 will be the target composition for our samples, the one that will be recreated. This Moon sample is the most studied of them all, [Mey09] Having been analyzed so deeply over the years without significant signs of disagreement on its composition or other properties provides an additional guarantee to the reliability of the data to be handled.

The following chapter gives an explanation about the process of mixing terrestrial basalt with additives in order to obtain a similar composition to 10084.

5 Synthesis of composition-oriented Lunar soil simulant (ITALUS)

Lunar simulants consist of materials found on Earth which have been combined nad processed in order to approximate the real Lunar soil in terms of composition, mechanical properties or other properties. [MCB94] For the production of the lunar simulant for the project, which will be referred as ITALUS (ITA Lunar Simulant), it is necessary to analyze in the first place the lunar simulants that exist nowadays.

5.1 Lunar simulants in existence

Lunar soil simulants have been produced on Earth since decades. Minnesota Lunar Simulant 1 (MLS-1), for instance, was produced by the University of Minnesota and resembles the chemistry of the Apollo 11's lunar sample 10084, taken at the Mare Tranquillitatis. [BS05] It comes from a basalt sill of an abandoned quarry in Minnesota, and contains a high content of Titanium. However, the most used and well-known simulant is the Johnson Space Center 1 (JSC-1) and its variations, JSC-1AF (fine), JSC-1A and JSC-1AC (coarse). NASA received 14 metric tons of JSC-1A and a ton of fine and coarse simulants extracted from the Merriam Crater. [SCS05] These simulants were also available for commercial sale by Orbitec until recently. This company currently sells only Martian simulant [www14a], but other companies have been able to emulate JSC-1A and sell it to the public, like ZAP. [www16g]

Five of the most common simulants and their composition by weight are comprised in the Table 5.1:

Table 5.1: Composition by weight of	common	lunar	simulants.	LOI:	Loss	of	ignition.
[MCB94] [SCS05] [ZWL05]							

	JSC-1 (mean of 3)	JSC-1A	CAS-1	FJS-1	MKS-1
SiO ₂	47,71 %	46,67 %	49,24 %	49,14 %	52,69 %
Fe ₂ O ₃	3,44 %	3,41 %	3,09 %	4,77 %	4,78 %
Al ₂ O ₃	15,02 %	15,79 %	18,52 %	16,23 %	15,91 %
MgO	9,01 %	9,39 %	7,32 %	3,84 %	5,41 %
CaO	10,42 %	9,90 %	4,11 %	9,13 %	9,36 %
Na₂O	2,70 %	2,83 %	3,69 %	2,75 %	1,90 %
Cr ₂ O ₃	0,04 %		0,01 %	n.d.	n.d.
TiO ₂	1,59 %	1,71 %	1,87 %	1,91 %	1,01 %
K ₂ O	0,82 %	0,78 %	1,38 %	1,01 %	0,58 %
FeO	7,35 %	5,75 %	8,26 %	8,30 %	7,50 %
MnO	0,18 %	0,19 %	0,19 %	0,19 %	0,22 %
P ₂ O ₅	0,66 %	0,71 %	1,28 %	0,44 %	0,14 %
SO ₃			0,03 %	n.a.	n.a.
CI					
LOI	0,71 %		1,02 %	0,43 %	0,50 %

JSC-1 (mean of 3) is the average composition of JSC-1A in its three variants: coarse, fine and regular. JSC-1A is the common JSC-1 composition (Figure 5.1). CAS-1 is a lunar simulant developed by the Chinese Academy of Sciences and based on the material of the Longgang volcanic cluster in Jillin. FJS-1 and MKS-1 are Japanese lunar simulants. It is a fact that Lunar simulants are used commonly in the space agencies all over the world for their tests, and most of them try to obtain their own material in order not to depend on other parts for their experiments.



Figure 5.1: A sample of about 5ml of JSC-1A by Arnold Reinhold [www16i]

5.2 Selection of base material

The chosen procedure for this project, as mentioned before, is only intended to emulate the lunar soil by weight composition and other analogies will not be directly taken into account.

The first step and objective of this thesis is the production of this synthetic simulant out of commercial materials, such as basalt powder or fibers, together with additives in the form of pure oxides. As a target figure, the base material of this simulant should not fall behind the 70% of the overall weight, which implies that the base material should be as close as possible to the target composition for a better transformation into simulant. Terrestrial basalt is a reasonable option for the starting base material, as it is a volcanic material similar in many aspects to the volcanic materials of the Moon. If this mixing process is realizable, a material with a significant chemical similarity to the Moon soil would be able to be produced from Earth materials and serve as a simulant for several projects of the kind.

The commercial basalt that has been chosen in this case is the Kammeny Vek's continuous basalt fiber, Basfiber®. The reasons behind this choice are the availability of the material at the institute and the positive feedback from other users. These fibers do not require special equipment or technologies for their processing, they are done through pultrusion and filament winding and their chemistry has been characterized successfully [www16m]. Its composition by weight is as shown in the Table 5.2:

Table 5.2: Composition by weight (%wt) of Kammeny Vek's Basfiber® [DC09]

	%wt
SiO2	55,69 %
Fe ₂ O ₃	10,80 %
FeO	0,00 %
Al_2O_3	15,44 %
MgO	4,06 %
CaO	7,43 %
Na₂O	2,40 %
TiO ₂	1,23 %
K ₂ O	1,51 %

The target Moon sample composition (10084) has some strong differences with the composition of commercial basalt fibers (Kammeny Vek's continuous basalt fiber), which are depicted in Table 5.3:

%wt	SiO ₂	Fe ₂ O ₃	FeO	Al ₂ O ₃	MgO	CaO	Na ₂ O	TiO ₂	K ₂ O
Basfiber®	55,69	10,80	0,00	15,44	4,06	7,43	2,40	1,23	1,51
Moon sample 10084	42,16	0,00	15,34	13,60	7,76	11,94	0,47	7,75	0,16

Table 5.3: Composition by weight (%wt) of 10084 and basalt fibers. [Mey09] [DC09]

The use of a commercially available basalt product was important for the synthesis of an inexpensive and easily producible simulant, so a process has been developed in order to modify the Basfiber® into a product with a closer chemical composition to the moon sample 10084.

5.3 Selection of additives

The basic procedure in summary is the concoction of basalt (in this case, Basfiber®) with other compounds in order to modify the overall composition by weight. The use of certain oxides and compounds need some considerations and restrictions. These assumptions have to be explained and taken into consideration, so that the similarity in composition is kept, especially in the overall behavior of the viscosity-temperature chart.

The first step is however to determine which additives should be added *a priori*. In Table 5.4 the composition differences between 10084 and Basfiber® are shown, and also the oxides whose concentration on Basfiber® should be increased (Incr.) or decreased (Decr.) in order to resemble 10084 are indicated:

Table 5.4: Oxides whose concentration should increase or decrease in the combination process.

%wt	SiO ₂	Fe ₂ O ₃	FeO	Al ₂ O ₃	MgO	CaO	Na ₂ O	TiO ₂	K ₂ O
Basfiber®	55,69	10,80	0,00	15,44	4,06	7,43	2,40	1,23	1,51
Moon sample 10084	42,16	0,00	15,34	13,60	7,76	11,94	0,47	7,75	0,16
	Decr.	Decr.	Incr.	Decr.	Incr.	Incr.	Decr.	Incr.	Decr.

The interpretation of Table 5.4 makes clear that FeO, MgO, CaO and TiO₂ should be the additive materials, and that they should be included in the mixture in the correct proportion to make the rest of oxides decrease their concentrations. The moon sample also contains MnO and P_2O_{51} , which are not shown in the Table 5.4. Their concentra-

tions are fairly low (0,2% and 0,05%), so they will not be considered as potential additives in this simulant.

The selection or discard of each of these materials as an additive, given their characteristics, availability and limitations will now be discussed.

5.3.1 Iron (II) oxide (FeO)

The most common compounds with iron are FeO and Fe₂O₃. However, FeO in its pure form is very expensive (21 €/kg) [www16l]. Such a price is not sustainable for the purposes of this project, as it would rise the overall cost of making the simulant. Another problem related to the use of FeO is the fact that for it to stay in its original form, special working conditions involving vacuum would be needed, thus increasing the complexity of the process and increasing the costs once more. Due to these reasons, FeO will not be used in this project.

Iron (II) oxide is however very abundant in the lunar sample 10084. As shown in Table 5.4, FeO constitutes the 10,80 % of the weight of the lunar sample, and it is the third most important compound, after SiO_3 and Al_2O_3 .

In the first place, FeO is an unstable compound when exposed to an oxygen rich environment, such as Earth's atmosphere, as the following chemical reaction takes place:

$$4 FeO + O_2 \rightarrow 2 Fe_2O_3$$
 (5.1)

This means that in a habitat on the Moon, which will be pressurized with a breathable atmosphere, the iron (III) oxide would oxidize. Simulant prepared with FeO would also undergo the same process, yielding a rich iron (III) oxide, a much less expensive material. If Fe_2O_3 were used in the first place as an additive, this oxidation could be spared and the same final stable composition would be the same. The price of production would also decrease with the usage of a common and more economical material [www16n]. Fe_2O_3 has a cost of $15 \in /kg$, compared to the $21000 \in /kg$ of pure FeO. The first alternative is, therefore, to use Fe_2O_3 instead of FeO.

Another possible alternative is to use Fe₃O₄. Iron (II,III) oxide, also known as magnetite, has both of the iron oxidation states and is also inexpensive. [www16q]

In order to do the testing as complete as possible, both alternatives will be put into practice and so, two types of simulant will be synthetized:

- The sample with Fe₂O₃, which will be referred as ITALUS-1.
- The sample with Fe₃O₄, which will be referred as ITALUS-2.

The composition of these two simulants and their ability to produce fibers will be studied in the following parts of the projects, their results being analyzed independently and later compared.

5.3.2 Magnesium oxide (MgO) and Calcium oxide (CaO)

The addition of both oxides independently is possible, as both materials are easily available. However, to reduce the amount of additives and ease the mixing process, a fact about the two can be considered.

The concentration of MgO and CaO influence the viscosity-temperature curves. In Figure 5.2 (MgO) and Figure 5.3 (CaO), the effect of increasing their amount by 1% with respect to the overall composition is shown to be similar, as both oxides lower the viscosity and the fiberizing temperature window. The calculation of the viscosity-temperature charts will be explained in 6.1.

Because of this, some considerations will be taken:

On the first place, the amount of MgO and CaO in the simulant samples will be counted together as a block. The simulants will try to have the same amount of combined MgO and CaO as the lunar sample 10084 has, but there will not be independent adjustment for both oxides, just as a whole.

On the second place, only one of these oxides will be required as an additive. As the Moon sample 10084 contains more CaO than MgO by a factor of 1,53:1, the preferred material to use will be CaO.

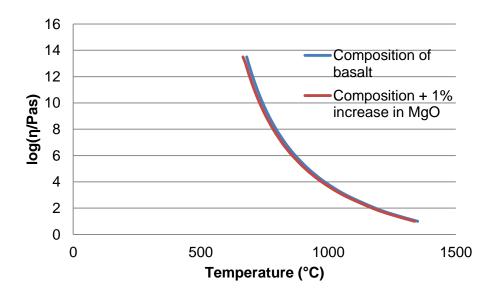


Figure 5.2: Viscosity-temperature chart for Basfiber® and Basfiber® with an increase in MgO from 4.06% to 5.06% by weight.

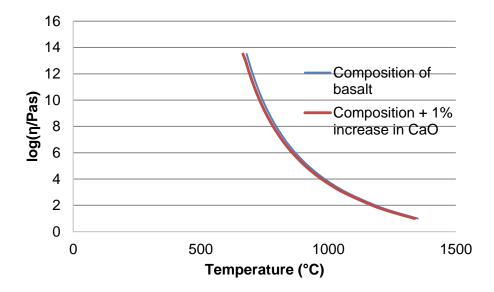


Figure 5.3: Viscosity-temperature chart for Basfiber® and Basfiber® with an increase in CaO from 7,43% to 8,43% by weight.

A last consideration about this section involves another simplification of the additive obtainment. Instead of using Calcium oxide in its pure form, CaCO₃ will be used. Calcium carbonate is a common compound found on rocks, largely studied, really inexpensive and available worldwide. [www16p]

For its use as an additive, however, it is important to consider how to transform it into CaO. Calcium carbonate can undergo a thermal decomposition into Calcium oxide and Carbon dioxide if subjected to a temperature of 850 °C: [Hil68]

$$CaCO_3 \to CaO + CO_2 \tag{5.2}$$

Pure Calcium oxide, if not slaked with water, is not stable when cooled, and reacts spontaneously with CO2 from the air. Therefore, and as the samples will undergo high temperatures for its mixing and later fiberization, it is equally apt to use Calcium carbonate, as it will decompose into Calcium oxide and the volatile Carbon dioxide will be emitted away from the sample. 560 g of CaO are contained in 1 kg of CaCO₃ and this will be considered in the further calculations.

The two variants of simulants that will be prepared, ITALUS-1 and ITALUS-2, will have the same amount of this additive each.

5.3.1 Titanium (IV) oxide (TiO₂)

Titanium (IV) oxide is an easily available material, widely used as a pigment under the name Titanium white or Pigment White 6, with applications in paint, sunscreen and even food coloring. It can be found in its pure form for a price of 23,10 €/kg. [www16o]

Increasing the content of TiO₂ is very relevant for the composition resemblance of ITALUS to the lunar sample 10084, as its percentage by weight has to increase from 1,23% to around 7,75%. This high concentration of titanium oxide is the reason why basaltic rocks and soil of the Sea of Tranquility are usually referred as high titanium basalts. Further studies involving the spectroscopic signature over this lunar region confirm the presence of titanium rich soil all over the mare. [DPI10] Titanium (IV) oxide is therefore a key additive to any simulant trying to approach the Mare Tranquillitatis average composition.

The two variants of simulants that will be prepared, ITALUS-1 and ITALUS-2, will have the same amount of this additive each.

5.4 The mixing process

After determining the materials that will give shape to the lunar simulants ITALUS-1 and ITALUS-2 (Table 5.5), the proportion of each element was calculated in order to reduce the deviation from the real sample.

Table 5.5: Constituents of ITALUS-1 and ITALUS-2

Simulant	Base material	Additives					
ITALUS-1	Basfiber®	Fe ₃ O ₂	CaCO ₃	TiO ₂			
ITALUS-2	Basfiber®	Fe ₄ O ₃	CaCO₃	TiO ₂			

The estimation of the proportion of each constituent has been determined with the use of tables. The following data is displayed first table type (Table 5.6, Table 5.10):

- Composition (wt%) of the base material
- Composition (wt%) of each of the additives
- Desired amount (%) of base material and additives for the mixture (input)
- Resultant composition (wt%) that a mixture with the desired setting would have (output)
- Target composition (wt%) of the lunar sample 10084
- Overall deviation from target composition $(wt\%_{resultant} wt\%_{target})$

Whereas in the second table type the following comparisons are shown (Table 5.7, Table 5.11):

- Concentration of SiO2 in the resultant and the target composition (wt%)
- Concentration of TiO2 in the resultant and the target composition (wt%)

- Combined amount of iron oxides for resultant and target composition (wt%)
- Combined amount of CaO+MgO for resultant and target composition (wt%)
- Deviation from target's composition $(wt\%_{resultant} wt\%_{target})$

The base material is made of 8 different compounds. If all of them had to be adjusted to the Lunar sample values, also 8 additives would be needed for the algebraic system to be solvable. With less additives, like the present case of 3, the system is underdetermined, which either has no solution or infinitely many solutions. Different boundary conditions, constraints and matrix sizes, with extra additives, have been tested in order to get a valid solution, but the results were inconsistent. Because of this, the used method was an approximate one, which did not intend to give a resulting exact composition to the lunar sample, but to adjust the main compounds (SiO₂, iron oxides, CaO, TiO₂) as accurate as possible. That is why the values for these compounds are presented in Table 5.7and Table 5.11.

5.4.1 Mixing process of ITALUS-1

The procedure is summarized in the following Table 5.6 and Table 5.7.

Table 5.6 shows us that the minimum allowed content of the base material, 70%, is needed in order to get a satisfactory resemblance with the lunar sample. The additives make up to the 30% of the mixture, of which 18% are Calcium carbonate, 7.5% are Iron (III) oxide and 4.5% are Titanium (IV) oxide. For the deviations, the values for the Iron oxides, Magnesium oxide and Calcium oxide are not shown as they are not relevant separately, and their combined values are depicted in the Table 5.7. Also CO₂ is not shown, as it will leave the mixture.

Table 5.7 also displays the most relevant compounds of the mixture, and their deviation from the target composition. There is a lower concentration of SiO2 than in the lunar sample, but the difference is acceptable. TiO2 is still not as high as in the target composition, but it has been raised considerably from the initial 1,23%. The figures for the combined MgO+CaO and FeO+Fe2O3 are satisfactory, especially for the iron oxides.

Table 5.6: Mixing process for ITALUS-1

wt%	Basfiber®	CaCO ₃	Fe₂O3	TiO ₂	Resultant mixture	Target sample	Deviation
SiO ₂	55,69				38,98	42,16	-3,18
Fe ₂ O ₃	10,80		99		14,99	0,00	
Al ₂ O ₃	15,44				10,81	13,60	-2,79
MgO	4,06				2,84	7,76	
CaO	7,43	55,50			15,19	11,94	
Na ₂ O	2,40				1,68	0,47	1,21
TiO ₂	1,23			99	5,32	7,75	-2,43
K ₂ O	1,51	0,40			1,13	0,16	0,97
FeO					0,00	15,34	
MnO					0,00	0,20	-0,20
P ₂ O ₅					0,00	0,05	-0,05
CO ₂		44,10			7,94		
SUM	98,56	100,00	99,00	99,00	98,87	99,43	
	Content of	base mater (wt%)					
	70,0	18,0	7,5	4,5			

Table 5.7: The most relevant compounds of the mixing process and their deviation from the target sample composition

	Resultant mixture	Target sample	Deviation
SiO ₂	38,98 %	42,16 %	-3,18 %
TiO ₂	5,32 %	7,75 %	-2,43 %
MgO+CaO	18,03 %	19,70 %	-1,67 %
FeO+ Fe ₂ O ₃	14,99 %	15,34 %	-0,36 %

CO2 will be lost after heating the sample, but it is shown in the Table 5.6 for the consistency of the values. It also indicates that at least 7,94% of the mass of the mixture will be lost when heated. This will make the other compounds rise their overall proportion in weight (Table 5.8)

Table 5.8: ITALUS-1 composition (wt%) before and after being heated. The deviations of the most relevant components appear bold.

	ITALUS-1 before heated	ITALUS-1 after heated	Target sample	Deviation	Dev. of combined pairs
SiO ₂	38,98%	42,87%	42,16%	0,71%	
Fe ₂ O ₃	14,99%	16,48%	0,00%	16,48%	1,14%
FeO	0%	0%	15,34%	-15,34%	1,1470
Al_2O_3	10,81%	11,89%	13,60%	-1,71%	
MgO	2,84%	3,13%	7,76%	-4,63%	0,13%
CaO	15,19%	16,70%	11,94%	4,76%	0,1370
Na ₂ O	1,68%	1,85%	0,47%	1,38%	
TiO ₂	5,32%	5,85%	7,75%	-1,90%	
K ₂ O	1,13%	1,24%	0,16%	1,08%	
CO ₂	7,94%	0%			

As it is noticeable, the loss of CO₂ is very convenient. All deviations have shrunk except for a slight rise for the sum of iron oxides, and none of the most relevant compounds surpasses now the 2 %. The adjustment to the target sample has been enhanced due to this effect. This heating process needed for the thermal decomposition of Calcium carbonate is also needed for fusing together the base material (fibers) with the additives (powder). The melted mixture is cooled down in the form of glassy frites, which are necessary for the posterior fiberization of the simulant.

Three batches with 75 g each will be prepared. The needed mass of each component is shown in the Table 5.9:

Table 5.9: Mass of base material and additives needed for ITALUS-1.

	wt%	Mass (g)
Basfiber®	70,0%	52,5
CaCO ₃	18,0%	13,5
Fe ₂ O ₃	7,5%	5,625
TiO ₂	4,5%	3,375
Total	100,0%	75,00

The physical mixing of the components is shown in the following images, Figure 5.4 and Figure 5.5.



Figure 5.4: Base material and additives for a 75 g ITALUS-1 sample. Additives



Figure 5.5: Three batches of ITALUS-1 before melting

Each of the samples was prepared with 52,5 g of Basfiber® and 22,5 g of additives. The resulting mixture is rather inhomogeneous, consisting of segments of basalt fibers and powder. The batches from ITALUS-1 and ITALUS-2 have been melted, the Carbon dioxide from the Calcium carbonate has been released and the mixture was converted into more homogeneous glass frits, more suitable for the posterior process of fiberization in the spinning machine.

5.4.2 Mixing process of ITALUS-2

The procedure is summarized in the following Table 5.10 and Table 5.11.

From Table 5.10 it can be indicated that the minimum allowed content of the base material, 70%, is needed in order to get a satisfactory resemblance with the lunar sample. The additives also make up to the 30% of the mixture, of which 18% are Calcium carbonate, 7.5% are Iron (III) oxide and 4.5% are Titanium (IV) oxide. For the deviations, the values for the Iron oxides, Magnesium oxide and Calcium oxide are not

shown as they are not relevant separately, and their combined values are depicted in the Table 5.11. Also CO₂ is not shown, as it will leave the mixture.

In the Table 5.11 the most relevant compounds of the mixture are displayed, and their deviation from the target composition. There is a lower concentration of SiO₂ than in the lunar sample, but the difference is acceptable. TiO₂ is still not as high as in the target composition, but it has been raised considerably from the initial 1,23 %. The figures for the combined MgO + CaO and FeO + Fe₂O₃ + Fe₃O₄ are satisfactory, especially for the iron oxides.

Table 5.10: Mixing process for ITALUS-2

wt%	Basfiber®	CaCO3	Fe2O3	TiO2	Resultant mixture	Target sample	Deviation
SiO ₂	55,69				38,98	42,16	-3,18
Fe ₂ O ₃	10,80				7,56	0,00	
Al ₂ O ₃	15,44				10,81	13,60	-2,79
MgO	4,06				2,84	7,76	
CaO	7,43	55,50			15,19	11,94	
Na ₂ O	2,40				1,68	0,47	1,21
TiO ₂	1,23			99	5,32	7,75	-2,43
K ₂ O	1,51	0,40			1,13	0,16	0,97
Fe ₃ O ₄			97		7,28		
FeO						15,34	
MnO					0,00	0,20	
P ₂ O ₅					0,00	0,05	-0,20
CO ₂		44,10			7,94	·	-0,05
SUM	98,56	100,00	99,00	99,00	98,87	99,43	
	Content of b	nase mater	ial and ad				

Content of base material and additives (wt%)

70,0 18,0 7,5 4,5

Table 5.11: The most relevant compounds of the mixing process and their deviation from the target sample composition

	Resultant mixture	Target sample	Deviation
SiO ₂	38,98%	42,16%	-3,18%
TiO ₂	5,32%	7,75%	-2,43%
MgO+CaO	18,03%	19,70%	-1,67%
Fe3O4+Fe2O3+FeO	14,99%	15,34%	-0,36%

 ${\rm CO_2}$ will be lost after heating the sample, but it is shown in the Table 5.10 for the consistency of the values. It also indicates that at least 7,94 % of the mass of the mixture will be lost when heated. This will make the other compounds rise their overall proportion in weight (Table 5.12)

Table 5.12: ITALUS-2 composition (wt%) before and after being heated. The deviations of the most relevant components appear bold.

	ITALUS-2 before heated	ITALUS-2 after heated	Target sample	Deviation	Dev. of combined pairs
SiO ₂	38,98%	42,87%	42,16%	0,78%	
Fe ₂ O ₃	7,56%	8,33%	0,00%	8,33%	
Fe ₃ O ₄	7,28%	8,02%	0,00%	-7,32%	1,01%
FeO	0,00%	0,00%	15,34%	-15,34%	
Al_2O_3	10,81%	11,89%	13,60%	-1,70%	
MgO	2,84%	3,13%	7,76%	-4,63%	0,16%
CaO	15,19%	16,70%	11,94%	4,79%	0,1070
Na ₂ O	1,68%	1,85%	0,47%	1,38%	
TiO ₂	5,32%	5,85%	7,75%	-1,89%	
K ₂ O	1,13%	1,24%	0,16%	1,08%	
CO ₂	7,94%	0%			

It can be observed that the loss of CO₂ is also convenient in this case. Most deviations have shrunk, and none of the most relevant compounds surpasses now the 2%. The adjustment to the target sample has been enhanced due to this effect. This heating process needed for the thermal decomposition of Calcium carbonate is also needed for fusing together the base material (fibers) with the additives (powder). The melted mixture is cooled down in the form of glassy frites, which are necessary for the posterior fiberization of the simulant.

But before that, the samples have to be prepared. For ITALUS-2, three batches with 75 g each will be prepared as well. The needed mass of each component is shown in the Table 5.13:

Table 5 13:	Mass of base	e material a	nd additives	needed for	ITALUS-2
I abic o. i o.	IVIAGO DI DAGI	, illatoliai a	na aaannvoo	IICCUCU IOI	11/12002

	wt%	Mass (g)
Basfiber®	70,0%	52,5
CaCO ₃	18,0%	13,5
Fe ₃ O ₄	7,5%	5,625
TiO ₂	4,5%	3,375
Total	100,0%	75,00

The physical mixing of the components is depicted in the following images, Figure 5.6 and Figure 5.7. The additives for ITALUS-2 are dark-colored.



Figure 5.6: Base material and additives for a 75 g ITALUS-2 sample



Figure 5.7: Three batches of ITALUS-2 before melting

5.5 The melting process

The next step in the synthesis of these lunar simulants consists on the melting of the samples. The main purposes of this process are:

- Trigger the Calcium carbonate thermal decomposition into Calcium oxide and Carbon dioxide
- Homogenization of the samples, incorporation of all the components in the mixture.
- Quenching of the mixture into glassy frits, which are more suitable to work with in the following fiberization process.

In order to execute this, an electrical oven was used. The oven allows the user to control the temperature of the samples by introducing the sequences of heating up and temperature holding required. ITALUS-1 and ITALUS-2 have some differences in composition and therefore different melting temperatures, however, the six samples (Figure 5.9) were successfully melted together by using a high temperature (1450 °C). The selected program for the electrical oven is shown in the following diagram. (Figure 5.8)

The has been heated up at a constant rate of 100 K/h until 300°C, then hold for an hour, followed by a second heating at a constant rate of 200 K/h which will bring the samples to a temperature of 1450°C. After the desired temperature is reached, the oven goes back to a holding state which, for this case, lasted for 5h25min. The whole process takes 11h10min. The selected temperature was estimated using the viscosity-temperature charts that will be revised later on.

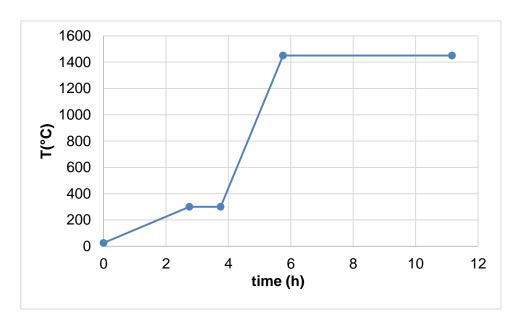


Figure 5.8: Program for the electrical oven: two heating phases and two holding phases

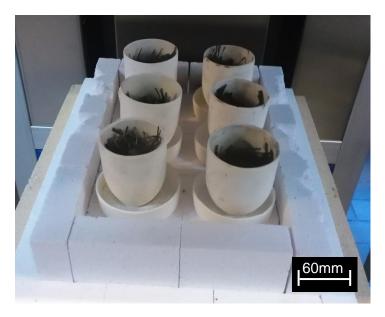


Figure 5.9: Batches prepared for melting

After the previous melting process occurs, the content of the batches has to be poured into cold water for the formation of frits. In Figure 5.10 it is depicted how this procedure was done:



Figure 5.10: Pouring the melted ITALUS-1 into water

The frits were dried for 24h after they were formed so that they lost the water. After doing so, it is possible to measure their weight properly and they will be prepared for the fiberization process.

The results of this stage are visually similar for both ITALUS-1 and ITALUS-2, but they will be presented independently. After the frits were done, however, both simulants lost due to the same causes:

- Loss of Carbon dioxide after Calcium carbonate decomposes, estimated to be of around 17,86 g.
- Loss due to material solidified in the alumina containers before being poured to water. (Figure 5.11) This loss is inherent to the procedure and partially irreversible, as trying to recover this material could add undesired pieces of alumina to the mixture, thus altering the composition of the sample.

The material was homogeneous, so a loss of it does not in reality affect the chemical composition of the samples.

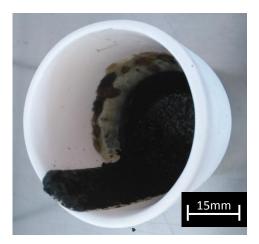


Figure 5.11: Material attached to the container vase

5.5.1 Melting process results for ITALUS-1

ITALUS-1's three batches had an initial mass of 225 g combined, but the mass after the frits were done was 146,7 g.

Figure 5.12 is a picture of a sample of the resulting frits:



Figure 5.12: ITALUS-1 frits

5.5.2 Melting process results for ITALUS-2

ITALUS-1's three batches had an initial mass of 225 g combined, but the mass after the frits were done was 145,5 g.

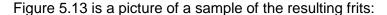




Figure 5.13: ITALUS-2 frits

The first objective of this project has been now accomplished. Two artificial lunar soil samples have been created, ITALUS-1 and ITALUS-2, which simulate the composition of 10084 in two different ways. If compared to Figure 4.1, ITALUS-1 and ITALUS-2 have now a chemical composition that simulates the real Moon samples. Some deviation from the original samples are inside an acceptable range of common variability of natural soil. The main deviation from 10084 is related to the grain size of the samples,

which is bigger than in 10084. With the adequate equipment, however, the samples can be grinded to the desired particle size and emulate another aspect of the original soil. Other characteristics and differences about the synthetic soil and the real one can be measured and analyzed for a better understanding of the crystallography, strength, magnetic properties, etc. that they may share.

6 Fiberization of the obtained lunar simulants

The second object of the thesis consists on the usage of ITALUS-1 and ITALUS-2 in ISRU testing. Given the resemblance in composition that these simulants have to the target lunar sample, testing their ability to form fibers may yield useful information about the feasibility of producing fibers out of lunar regolith from the Sea of Tranquility. Simulants are usually processed in accordance to the user's needs, mimicking the aspects that are more important for the development of their work, in this case, the composition was chosen as the determining characteristic.

The fiber forming consists on various phases: bushing, sizing, wending and post-processing. [PSG10] The basic idea of fiber formation consists on drawing off fiber from a crucible, a piece which allows the flow of melted glass in the direction of gravity, attenuating the filaments while doing so. In order for a fiber to be fiberized, the viscosity has to range from 30 to 100 Pas, and the required temperature to get such a viscosity shall be computed before the fiber forming process. Once this temperature range is reached, the bushing takes place, in which the compensation of gravity, friction and fluid mechanic terms allows the melted glass to move through a small hole, which gives the fiber defined diameter. The resulting fiber is winded in a strand and may be post-processed afterwards.

Given the previous information, to proceed with the fiberization it is important to firstly analyze and determine the temperature in which the simulants will get a suitable viscosity for fiber formation. This will be done through the study of the viscosity-temperature charts in the following section.

6.1 Fiberization window

The viscosity-temperature charts have been calculated in order to provide a view of the suitable temperatures for fiber formation in crucibles. [Pic10] [PSG10] The viscosity of a melted glass depends on the temperature and the chemical composition, so for each simulant a temperature-viscosity curve was calculated. The relevant data of the graphs consist on the temperatures in which the viscosity lies between 30 and 100 Pas, or between 1,5 and 2 in the logarithmic scale, as this viscosity window allows the formation of fibers through the crucible and, therefore, by controlling the temperature of the oven, the control the formation of fibers can also be controlled.

The software used for these simulations of viscosity is called ViscCalc, an spreadsheet based on the work of Fluegel *et al.* [FVE04] The program estimates the viscosity of industrial glasses based on their chemical composition with an error of 12°C given an input composition in mol%. The program is based on experimental data and there are composition ranges where the error is lower than 12 °C.. It also warns when the limits are exceeded for certain components. In the case of ITALUS-1, ITALUS-2 and all basalt-based compositions in general, the program exceeds the composition and interaction limits, as it was programmed to work with different kinds of glass, with low iron oxide content. The samples exceed these limits in TiO₂ AND Fe₃O₂.

An additional program was used for the conversion of weight percentages into mol%, also by Fluegel, called GlassViscCalc. [Flu07] Even if it is an updated version of the latter, it does not plot the results of the graph. The data, however, can be extracted and plotted in a separate spreadsheet. The results of the calculations and plotting for ITALUS-1 and ITALUS-2 are presented in the following sections.

6.1.1 Fiberizing window for ITALUS-1

The expected behavior of the viscosity with the temperature is charted in Figure 6.1, whose data points are shown in Table 6.1:

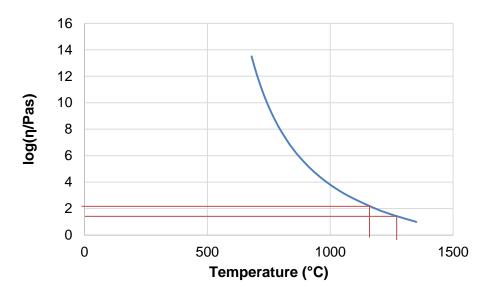


Figure 6.1. Viscosity-temperature chart for ITALUS-1 with the indicated fiberizing window

Table 6.1: Data for the viscosity-temperature chart of ITALUS-1. The fiberizing temperature window appears in bold

log(η/Pa*s)	T (ºC)
1	1350,2
1,5	1259
2	1184,4
3	1069,7
4	985,6
5	921,3
6	870,5
6,6	844,9
7	829,4
8	795,5
9	767
10	742,7
11	721,8
12	703,5
12,3	698,5
13	687,5
13,5	680,2

The sample surpassed the absolute maximum values allowed only for Titanium (IV) oxide and Iron (III) oxide. However, the experimental part proved that the calculation was accurate, as the actual working temperature was 1200 °C for the lower part of the crucible, and 1250 °C in the upper part, as seen in Figure 6.2



Figure 6.2: Working temperature in the lower part (left) and at the upper part (right) of the oven. ITALUS-1

6.1.2 Fiberizing window for ITALUS-2

The expected behavior of the viscosity with the temperature is charted in Figure 6.3, whose data points are shown in Table 6.2:

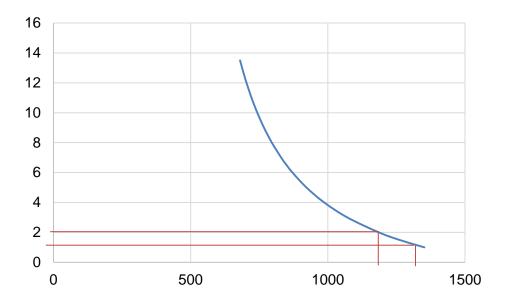


Figure 6.3: Viscosity-temperature chart for ITALUS-2 with the indicated fiberizing window

Table 6.2: Data for the viscosity-temperature chart of ITALUS-2. The fiberizing temperature window appears in bold

	,
log(η/Pa*s)	T (ºC)
1	1351,8
1,5	1260,6
2	1185,9
3	1070,9
4	986,6
5	922,1
6	871,1
6,6	845,4
7	829,9
8	795,8
9	767,2
10	742,8
11	721,8
12	703,5
12,3	698,4
13	687,4
13,5	680

The results for ITALUS-2 exceed the limits for Titanium (IV) oxide and Iron (III) oxide. This model has Iron (II,III) oxide together for the calculation, which is assumed to be Fe_2O_3 for the calculations. The integration of this component and its interaction with the rest of components is unknown, as the software does not allow this kind of oxide as an input. Instead, the sum of iron oxides has been introduced as just Iron (III) oxide, in order to calculate the viscosity-temperature curve. The results of this assumption for ITALUS-2 were confirmed by the fiberization experiments. The temperature range between 1220 °C and 1270 °C at the bottom and at the top of the crucible:



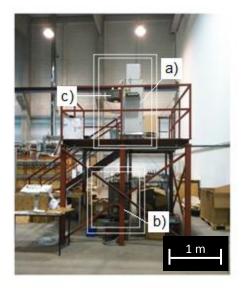
Figure 6.4: Working temperature in the lower (left) and at the upper (right) spinning machine's heaters. ITALUS-2

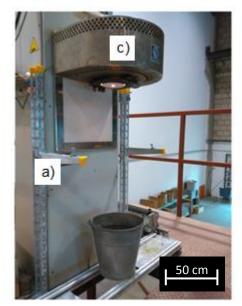
6.2 Fiberization process

The fiber forming, as mentioned before, consists on various phases: melting, sizing, winding and post-processing. [PSG10] The expected temperature window for fiberization has been determined.

In the bushing phase, the compensation of gravity, capillarity and thermo-fluid mechanic terms allows the melted glass to flow through a small nozzle. A difference of around 50°C between the upper and lower part of the crucible allows to near the pilot plant to the industrial one. The diameter of the resulting fibers is determined by the temperature, the nozzle diameter and the take-up velocity

The facilities used for this project are depicted in Figure 6.5:





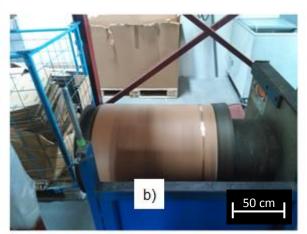


Figure 6.5: Spinning machine architecture. a) Electrical heater, controls, crucible. b)

Spinning applicator for welding. c) Location of the two electrical heaters and the crucible. The hole out of which fibers are drawn off can be appreciated.

After the batches reach the set working temperature (Figure 6.2, Figure 6.4), droplets will start to fall down from the crucible (Figure 6.6). For security and preservation of the facilities, a water container is placed under the open part of the crucible. When droplets fall on water, they solidify quickly and may explode due to the internal forces created and the thermal shock. When the temperature is adequate, the droplets draw with them an initial fiber, which can be drawn further by hand to the welding rotator and the spinning of fibers.

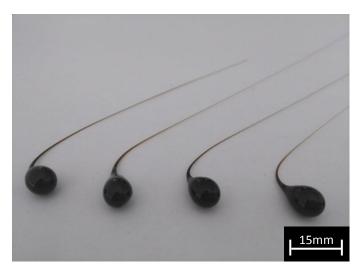


Figure 6.6: Some of the recovered droplets from ITALUS-1 with fiber formation

6.3 Results

The spinning process was successful for both ITALUS-1 and ITALUS-2. It was possible to spin 2 different samples of fibers from ITALUS-1 and 3 samples for ITALUS-2.

The fibers were later measured in order to study their width and obtain more data for the determination of fiberizing parameters. Longer spinning times, fiber spinning velocity and lower diameter are related to better fiberizing performance. A summary of the main aspects of the fiberization results is shown in Table 6.3, and the following pages contain a more detailed version of these results.

Table 6.3:	Summary	v of results	of the	fiberization	process
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Sample name	Temperature range (°C)	Spinning time	Continuity of production	Spinning velocity	Diameter (µm)
ITALUS-1 Probe 1	1200-1250	5 min	unstable	1,7 m/s	12,334
ITALUS-1 Probe 2	1200-1250	4 min	unstable	1,7 m/s	16,711
ITALUS-2 Probe 1	1220-1270	20 min	stable	1,7 m/s	17,453
ITALUS-2 Probe 2	1220-1270	13 min	stable	3,6 m/s	33,924
ITALUS-2 Probe 3	1220-1270	3 min	unknown	1,7 m/s	9,482

6.3.1 Resulting fibers for ITALUS-1

Two samples were produced in the experiments with ITALUS-1. The resulting fibers spun for 5 and 4 minutes before breaking and the spinning velocity was the same for both samples.

The most likely sources of error regarding of the early fracture of fibers were:

- The brittleness of the material.
- The rotator velocity, which may have pushed the fibers beyond their limit of tension.
- The blocking of the crucible nozzle with big particles.

The macroscopic and microscopic view of the probes 1 and 2 are exhibited in Figure 6.7 and Figure 6.8.

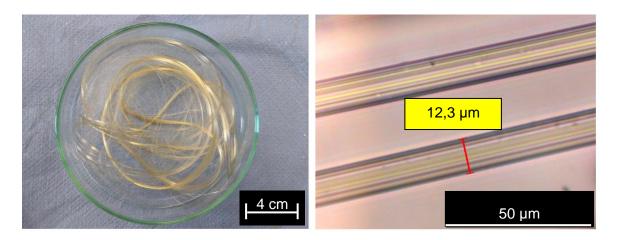


Figure 6.7: Macroscopic (left) and microscopic (right) view of the ITALUS-1 probe 1 resulting fibers. The measurement of diameter is appreciable.

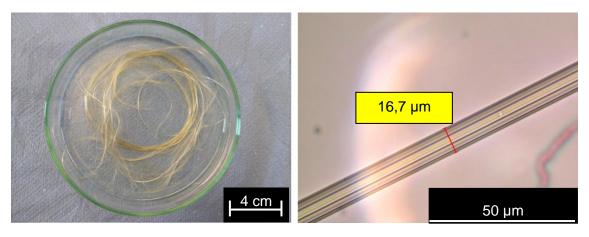


Figure 6.8: Macroscopic (left) and microscopic (right) view of the ITALUS-1 probe 2 resulting fibers. The measurement of diameter is appreciable.

6.3.2 Resulting fibers for ITALUS-2

Three samples of ITALUS-2 were produced. The first two probes were stable in the spinning at constant speed, whereas the last probe was stopped due to the lack of enough material in the crucible.

It is interesting that the probe 2, which was spun at a higher speed than probe 1, has a bigger diameter. This contradiction can be explained by the blocking of the crucible by non-melted particles or bubbles that are common in small spinning plants. These would have modified the allowed cross-section diameter. The excessive filling of the crucible can affect the process too, Partial solidification on the top can seal it and stop the flow of melted material.

The composition for probe 1 and probe 2 is expected to be entirely composed of ITALUS-2, as all the previous traces of material in the crucible were melted out. However, probe 3 was spun just after the ITALUS-1 probes, and it is not clear if it contains some of that other composition. This did not impede the fibers to form, as the composition of both simulants is very close, and are only different in the oxidation state of the iron. Sample three also has the smallest diameter of all the probes, even though the used speed was not the highest.

The macroscopic and microscopic view of these probes are exhibited in Figure 6.9, Figure 6.10 and Figure 6.11

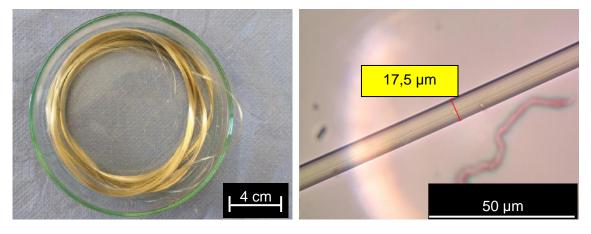


Figure 6.9: Macroscopic (left) and microscopic (right) view of the ITALUS-2 probe 1 resulting fibers. The measurement of diameter is appreciable.

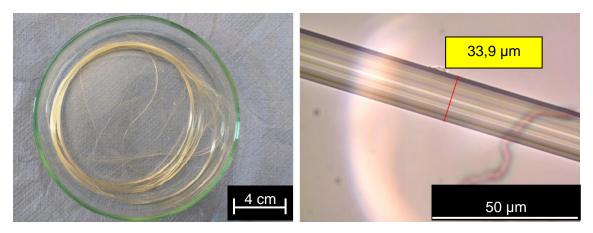


Figure 6.10: Macroscopic (left) and microscopic (right) view of the ITALUS-2 probe 2 resulting fibers. The measurement of diameter is appreciable.

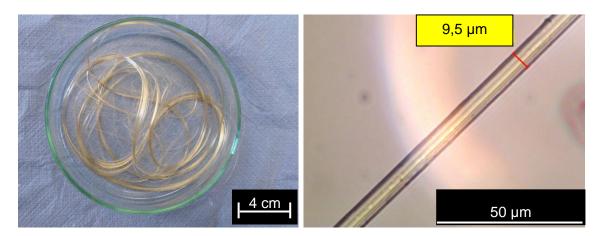


Figure 6.11: Macroscopic (left) and microscopic (right) view of the ITALUS-2 probe 3 resulting fibers. The measurement of diameter is appreciable.

7 Conclusion and outlook

Aim of this thesis was to give an answer to the following questions:

- 1. Is it possible to synthetize lunar soil using common commercial materials on Earth?
- 2. Is it possible to produce fibers with this simulant and, by extrapolation, with Moon regolith?

The first goal was accomplished as two different lunar simulants, ITALUS-1 and ITALUS-2, were produced. The synthesis of the materials needed a previous study of the Moon composition data to wholly understand the characteristics of the lunar surface material. This documentation helped in the correct selection of a target lunar sample whose composition was set as a target for the produced artificial soil. Commercially available materials were studied and chosen for the forming of this sample. However, a deeper insight in the data from lunar samples led to the diversification of the simulants to be produced, each one approaching the problematic differently. The simulants were produced by melting and quenching a mixture of basalt and other additives. Due to quenching, the mixtures kept an amorphous structure and glass frites were formed.

Once the simulants were fabricated, they were tested for the ISRU study of fiber production. The first step for this objective was to compute the temperature window in which the viscosity of the melted samples allows fiberization. For the determination of said data, simulation by software was used. The simulants were melted in a monofilament spinning machine, and fiber was drawn off the crucible. The process seemed to be more stable on the ITALUS-2 case than on the ITALUS-1, whose fibers fractured after some minutes of spinning. However, both simulants were able to be fiberized, and that positively fulfills the initial question. This thesis has proven the possibility of fiber formation from the lunar regolith and has provided a valid method and data about the procedures for its reproduction in future experimentation.

If the results for these simulants are extrapolated for the real case, multiple applications and uses for the fibers arise. It is expected that they show similar properties to basalt fibers, as they consist mostly of that material. The commercial basalt fibers have a tensile strength would lie between 2700 and 3200 MPa, with a tensile modulus from 85 to 95 GPa. [Tho13] Basalt fibers have higher mechanical properties and have a better chemical resistance in acid and alkali environments, they can work up to 580 °C.

Possible applications on the lunar surface include their use in the construction of underground habitats. Basalt fibers can be used as reinforcement of structures due to their high modulus of elasticity and their heat resistance. Bars made of basalt fibers can be produced for this application. Possible structural damaging of concrete columns can also be repaired by wrapping basalt fiber sheets, as shown in Figure 7.1, and that would serve as an excellent reparation material for lunar based stations or mines.



Figure 7.1. Renovation of concrete columns by wrapping basalt fiber sheets. [Tho13]

Another application involving the use of basalt as reinforcement material is their use in "Lunar pykrete". Pykrete is a composite material made primarily of water ice and wood pulp or sawdust. During the Second World War, Geoffrey Pyke proposed its use for the fabrication of ships, and it has been studied and perfectioned over time. Institutions like the Vienna University of Technology and Eindhoven University of Technology ingeneered a method to construct a pykrete-reinforced ice domes as can be observated in the Figure 7.2. [Bry11]



Figure 7.2: Pykete-reinforced ice dome built in Jukka, Finland, by the Eindhoven University of Technology. [Van14]

Short lunar basalt fibers would substitute sawdust in the Moon, and habitats would be built by spraying over ice domes. Permanently shadowed areas could keep the ice in solid state. The Moon contains water in solid state, which can be extracted and serve as the matrix for this composite materials. Pykrete could also be produced in smaller block for building other structures of for radiation shielding. In the field of construction, lunar fibers could also be used as insulation material in the form rock wool. All of these pykrete and construction applications can be extrapolated to the Martian case as well. Mars has water ice stored in its poles, a soil composition which could be suitable for fiber production and temperatures below the freezing point of water to keep this material solid. The study of fiber formation from Martian soil and its applications should be continued in future works.

Lunar fibers could also be used in reinforcement for the manufacturing of mechanical pieces, tools, pieces of furniture, interior of habitats coating and can also be braided into fabric for clothing inside the habitat and for astronaut suits.

However, in order to study the actual applications of the lunar basalt fibers, the characterization of the fibers should be more extensive. Real lunar soil samples are needed for this task to yield completely reliable data, as simulants are only an approximation. The gravity of the Moon, which is only 16% of Earth's gravity, would also affect the formation of fibers: A lower gravitational pull would decrease the hydrostatic pressure of the melted material in the crucible, and destabilize the equilibrium of forces needed for the flow. Therefore, new spinning machines and crucibles adapted to this reduced gravity should be studied and manufactured.

The lunar simulants produced in this thesis can be upgraded and refined or directly used for future projects. The first version of ITALUS-1 and ITALUS-2 kept the chemical composition of the moon sample 10084, but further mechanical processing could improve it and make it more attractive to the usual users of lunar simulants, which mostly look for simulated physical characteristics, size, shape, particle size and grain size distribution. These properties should be determined in future works, as well as the characterization of the fibers from ITALUS-1 and ITALUS-2. By producing a simulant matrix from the Moon, the characterization of composites with the fibres can be done as well, and the properties of these composite materials can be determined.

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9 Statement of academic honesty

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