CHARACTERIZATION AND EXPANSION BEHAVIOUR OF PERLITE

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by
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ABSTRACT

CHARACTERIZATION AND EXPANSION BEHAVIOUR OF PERLITE

Perlite is a silica-based organic glass. When it is instantaneously heated, it shows expansion by releasing a certain amount of water. Approximately 70% of perlite reserves in the world are found in Turkey. The porous structure of expanded perlite leads to use this material in many industrial areas; therefore, this is highly advantageous from the economic standpoint.

In this study, the expansion behaviours of perlite samples, which were taken from İzmir (Bergama Mine), Kütahya (Avdan Mine), and Erzincan (Mollatepe Mine) were both characterized and investigated in the laboratory. The instrumentation techniques, X-ray Diffraction (XRD), X-ray Fluorescence (XRF), Scanning Electron Microscopy (SEM), Thermogravimetric Analysis (TGA), and Optic Microscope were used to characterize the perlite. The determination of the amorphous and crystal structure of perlite, the chemical composition of perlite, the measurement of loss of mass, the morphological characterization of perlite, and the form of raw and expanded perlite samples were obtained by XRD, XRF, TGA, SEM and Optic Microscopy, respectively.

Four different heat treatments as 600 ˚C, 800 ˚C , 900 ˚C and 1000 ˚C and four different sizes 400-500 μm, 315-400 μm, 200-315 μm and 160-200 μm were conducted in the laboratory and the their bulk volumes and densities were compared before and after heat treatment. The perlite sample from Bergama 01 showed the largest expansion in all samples. As results of the analyses, the amount of crystal and water release in the perlite which had an influence on expansion process were consistent with the literature. The analyses were conducted until 1000 ˚C due to forming sinter above softening temperature. Additionally, the expanded perlite samples in the industrial furnace were compared with the expanded perlite samples in the laboratory more porous structure was observed.

As a conclusion, the perlite samples from the different regions of Turkey were characterized and analyzed in terms of expansion behaviour in this study.

ÖZET

PERLİTİN KARAKTERİZASYONU VE GENLEŞME DAVRANIŞI

Perlit silica bazlı volkanik bir camdır. Ani olarak ısıtıldığında içindeki bir miktar suyun açığa çıkmasıyla genleşme özelliği gösterir. Dünya üzerindeki perlit rezervinin yaklaşık %70 den fazlası Türkiyede bulunmaktadır. Genleşmiş perltin gözenekli yapısı sayesinde birçok endüstriyel alanda kullanımı mevcuttur. Bu da ekonomik açıdan oldukça avantajlıdır.

Bu çalışmada İzmir (Bergama Madeni), Kütahya (Avdan Madeni) ve Erzincan (Mollatepe Madeni)'den alınan perlit örnekleri karakterize edilip labratuvar ortamında genleşme davranışları izlendi. Karakterizasyon özelliklerini belirlemek için XRD, XRF, TGA, SEM ve Optik Mikroskobu kullanıldı. Perlitin amorf ve kristal yapısını belirlemek için XRD cihazı, perlitin kimyasal bileşiklerinin belirlenmesinde XRF cihazı, sıcaklıkla ağırlık kaybının ölçülmesinde TGA cihazı, morfolojik yapı hakkında bilgi alınması için SEM cihazı ve ham ve genleşmiş perlit örneklerinin görüntüleri için ise Optik Mikroskop kullanılmıştır.

Labrotuvar ortamında genleştirilen 600˚C, 800˚C, 900˚C ve 1000˚C sıcaklıklarda ve 400-500 μm, 315-400 μm, 200-315 μm ve 160-200 μm boyutlardaki perlit örnekleri sıcaklık öncesi ve sonrasındaki saf hacimleri ve yoğunlukları dereceli silindir ile ölçülüp genleşme oranları karşılaştırılmıştır. Tüm analizler sonucunda ise Bergama bölgesinden alınan Bergama 01 örneği en çok genleşmeyi sağladığı görülmüştür. Literatürle bağlantılı olarak genleşmeyi etkileyen perlitin içindeki Kristal miktarı ve su çıkışı yapılan analizler sonucu bu çalışmada tutarlı bulunmuştur. Softening pointin üzerindeki sıcaklıklarda sinterleme meydana geldiğinden analizler 1000 derece sıcaklığına kadar uygulanmıştır. Ayrıca Endüstriyel fırında genleştirilen perlit örneklerinin labratuvar ortamınkinden daha fazla gözenek oluşumu olduğu görülmüştür.

Bu çalışmada sonuç olarak Türkiye nin farklı bölgelerinden alınan perlit örneklerinin karakterizasyonları analiz edilerek genleşme davranışları izlenmiştir.

To my Mom and Dad…

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CHAPTER 1

INTRODUCTION

1.1. Perlite

Perlite is a natural volcanic glass of alumina-silicate composition with entrapped water which has resulted after quick cooling and solidification of volcanic lava. It is a volcanic rock, light gray, presented in Figure 1.1. The name which means pearl is derived from ‘perle’. When some type of perlite crack small spheres such as pearl luster is formed as a result perlite was given as name (Orhun, 1969). Perlite for the first time was expanded in 1940 in Las Vegas to get experience in a furnace and was used to obtain building plaster (MTA, 1985). It is estimated that perlite was mined from Picket Post Mountain located in Surerior (Arizona) in 1941 (MTA, 1985).

Figure 1.1. Raw perlite.
(Source : The European Commission under the 7th Framework Programme, April 2013)

1.1.1. Chemical and Physical Properties of Perlite

Chemically, it is an inorganic material which is generally composed of amorphous silica with 12-18 % aluminum oxide, smaller amounts of the oxides of potassium and sodium, less amounts of iron, magnesium, calcium, and titanium (Ciullo, 1996). Elemental analysis of perlite from China is given in Table 1.1 as an example of the compositional variations one may encounter for this material. The distinguishing feature which set apart from other volcanic glass is that when rapidly heated perlite to temperatures about 760-980 ˚C, it expands 4-20 times its original volume whose generic name is amorphous volcanic alumina-silicate rock (Daniel Maxim, Niebo, & E. McConnell, 2014). If perlite was heated slowly, water will be released gradually and no expansion occurs (Aguilar-Garib, García-Onofre, Ortiz, & Valdez-Nava, 2013). After heat treatment, it is converted to low density and porous structure, and changes its color from gray to white, presented in Figure 1.2. Perlite which is defined as having 2-5 % water, occurs around the world in glassy, rhyolitic, high-silica volcanic rocks less than 60 million years old (Barker & Santini, 2006). The physical properties of perlite are indicated in Table 1.2.

Figure 1.2. Expanded perlite.
(Source : The European Commission under the 7th Framework Programme, April 2013)

Table 1.1. Chemical Analysis of perlite from China.
(Source : (a) Uemura, et al., 1999, (b) Jing, Fang, Liu, & Liu, 2011)

Chemical Composition	**SiO_2**	**Al_2O_3**	**K_2O**	**Na_2O**	**CaO**	**Fe_2O_3**	**MgO**	**TiO_2**	**FeO**	**MnO**
Perlite China (a) (Uemura, et al., 1999)	72,70%	13,56%	4,25%	3,38%	0,66%	0,83%	0,11%	0,08%	-	-
Perlite China (b) (Jing, Fang, Liu, & Liu, 2011)	72,93%	12,9%	5,30%	2,57%	0,76%	0,53%	0,16%	-	0,18%	0,06%

Table 1.2. Some typical physical properties of perlite.
(Source: Perlite Institute, 2011)

Color	Gray, White (expanded), Black
Reflective Index	1.5
pH (of water slurry)	6.5-8.0
Free Moisture (max.)	0.5 %
Bulk Density (lose weight)	Expanded : 32-400 kg /m^3 Crude Ore : 960/1200 kg/m^3
Mesh Sizes	Avaible as desired; 4-8 mesh and finer
Softening Point	1600-2000 ˚F - 871-1093 ˚C
Fusion Point	2300-2450 ˚F - 1260-1343 ˚C

1.1.2. The Reserves of Perlite

The production of perlite around the world is that in descending order of production, Turkey, Greece, and The United States, 41%, 26%, %18, respectively. At least since 2008, in Greece and Turkey significantly more perlite production is carried out from the United States (U.S. Geological Survey, 2010-2011-2012-2013-2014-2015-2016). When looked around the world, the reserve of perlite is more in Turkey. The amount of perlite reserve is considerably higher than the world; show us that it can contribute substantially to the economy of Turkey.

World perlite reserves are estimated to be around 8 billion tons (Gürtürk, Oztop, & Hepbaslı, Energy and Exergt Assessments of a Perlite Expansion Furnace in a Plaster Plant, 2013) and 5.7 billion tons of it exist in Turkey (Industrial Raw Materials Coordinator Mustafa Kırıkoğlu, MİGEM (General Directorate of Mining Affairs), in Turkey), corresponding to more than about 70% of the world reserves.

Table 1.3. Turkey of Perlite Reserves.
(Source : MİGEM, 2007-2013)

Source of Perlite	**Reserves (Tons)**	**Percentage %**
Kars-Sarıkamış	2.043.000.000	33.9%
Van-Erciş	1.400.000.000	23.2 %
Bitlis-Adılcevaz ve Tatvan	940.000.000	15.6 %
Nevşehir-Derinkuyu-Acıgöl	800.000.000	13.3 %
Erzurum-Pasinler	386.824.000	6.4%
Çankırı-Orta-Kalfat	128.200.000	2.1%
İzmir-Bergama-Cumaovası-Foça	88.000.000	1.5%
Erzincan-Mollatepe	71.500.000	1.2%
Ankara-Çubuk	51.000.000	0.8%
Ankara-Kızılcahamam-Güvem	31.500.000	0.5%
Balıkesir-Şavaştepe	26.000.000	0.4%
Balıkesir-Sındırgı	21.206.000	0.4%
Manisa-Saruhanlı	17.700.000	0.3%
Kütahya-Avdan	11.500.000	0.2%
Ankara-Kızılcahamam-Çamkoru	8.000.000	0.1%
Ankara-Kızılcahamam-Korkmazlar	3.700.000	0.1%
Total	6.028.130.000	100%

Table 1.3. shows the perlite reserves in Turkey. Compared to the world, it is seen that the perlite reserve in Turkey is quite high. Perlite, therefore, is an important mineral for Turkey.

1.1.3. Applications of Expanded Perlite

Perlite has many different uses. It has applications in sandblasting, foundry and steel industries, special casting sand, metal finishing, silica source (Perlite Institure, Applications of Perlite, 2009). It is generally used in constructional and also used in ceramic industry (ceramic floor and wall tiles), in food industry, chemical industry, and in agriculture (Fiat, Lazar, Baciu, & Hubca, 2010). After the perlite is expanded, becomes lighter and more porous thus it is more preferable economically. It also provides a positive contribution to both the country's economy and the global warming. So, the use of expanded perlite is quite comman. Table 1.4. shows that general applications of expanded perlite.

Table 1.4. Applications of Expanded Perlite.
(Source : Perlite Institure, Applications of Perlite, 2009)

Ambient and Low Temperature Insulation Roof decking, concrete floor fill, concrete blocks, perlite asphalt, bituminized perlite, core filler in wallboards, cavity wall insulations, refrigeration plants, portable ice boxes, containers.	**Agricultural Additive and Supplement** Poultry litter supplement to reduce odor and moisture adsorbent, animal feed anti-caking agent and filler, carrier for nutrients and medicines.	**Medium to High Temperature Insulation** Boiler covering in quilted mattresses and in hard setting compositions, pipe covering in coaxial tubes, compression molded pipe half sections, pour-in pipe insulation.
High Temperature Insulation Foundry cores and molds, ovens, crucible topping.	**Oil Well Treatments** Oil well cementing and low density mud	**Fireproofing** Fire insulation in safes, rooms, doors, chimney linings.
Acoustic Plasters, mortars, plaster boards, ceiling tiles and upper wall insulation, highway sound absorbing walls.	**Horticulture** Plant rooting, seed starting medium, growing medium, soil conditioner, seed coating, hydroponic, green roofs.	**Liquid Filtration** Beer, wine, edible oils, citric acid, sugar, oils, pharmaceuticals, fruit juices, glucose, chemicals, wort, swimming pool water, potable water, storm water runoff, bio diesel.

1.2. A Literature Review on Expanded Perlite

Perlite is known to expand abruptly when heated to a temperature range between 700-1200 ˚C, where this expansion behavior has been expressed to show similarities with the popping of popcorn. This causes expanded perlite to attain a porous structure and a lighter weight material. In this way, expanded perlite becomes softer and has more porous structure.

The perlite is expanded into a lightweight, versatile new material. Light with apparent density, low thermal conductivity, chemical stability, wide temperature range, and moisture absorption capacity is small, and non-toxic, odorless, fire, acoustic and other characteristics, widely used in many industrial sectors.

According to literature, Jing et al. were used to determine expansion properties of the perlite which is from Shangtianti in Xinyang, China (Jing, Fang, Liu, & Liu, 2011). It is important points that water content, preheating temperature, expanding temperature, calcination time and size of raw particles. They used a horizontal electric furnace for these expansion conditions Figure 1.3. shows the affect of preheating temperature on expansion volume.

Another literature, Kaufhold et al. were used perlite sample in Milos, Greece. They characterized the bulk material with different analysis method such as XRD, XRF, the micro-computed tomography etc (Kaufhold, et al., 2014). Figure 1.4. shows the XRD patterns with crystal componets and the chemical composition.

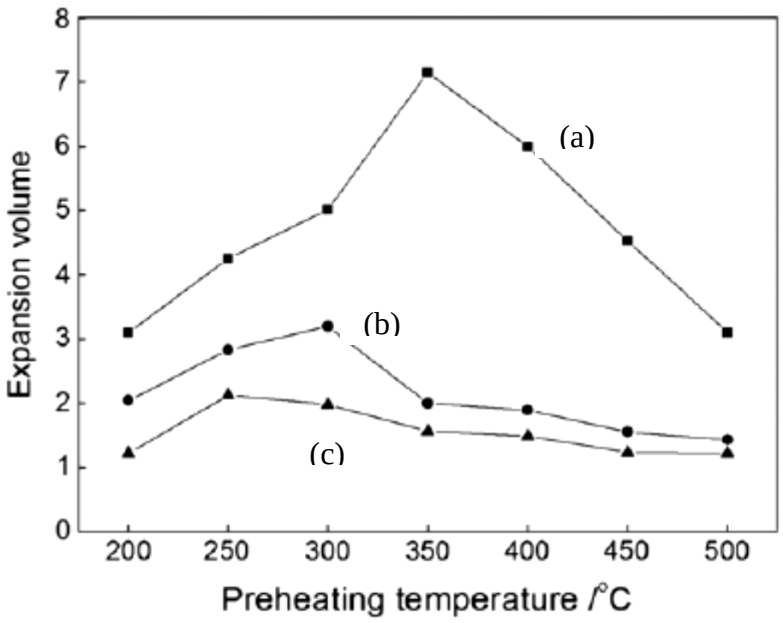


Figure 1.3. Expansion volume of perlite (China) with different size and preheating temperature (a) 420-840μm, (b) 150-178μm, (c) 100-123μm (preheating time is 10 s, expanding temperature is 1200 ˚C.
(Source : Jing, Fang, Liu, & Liu, 2011)

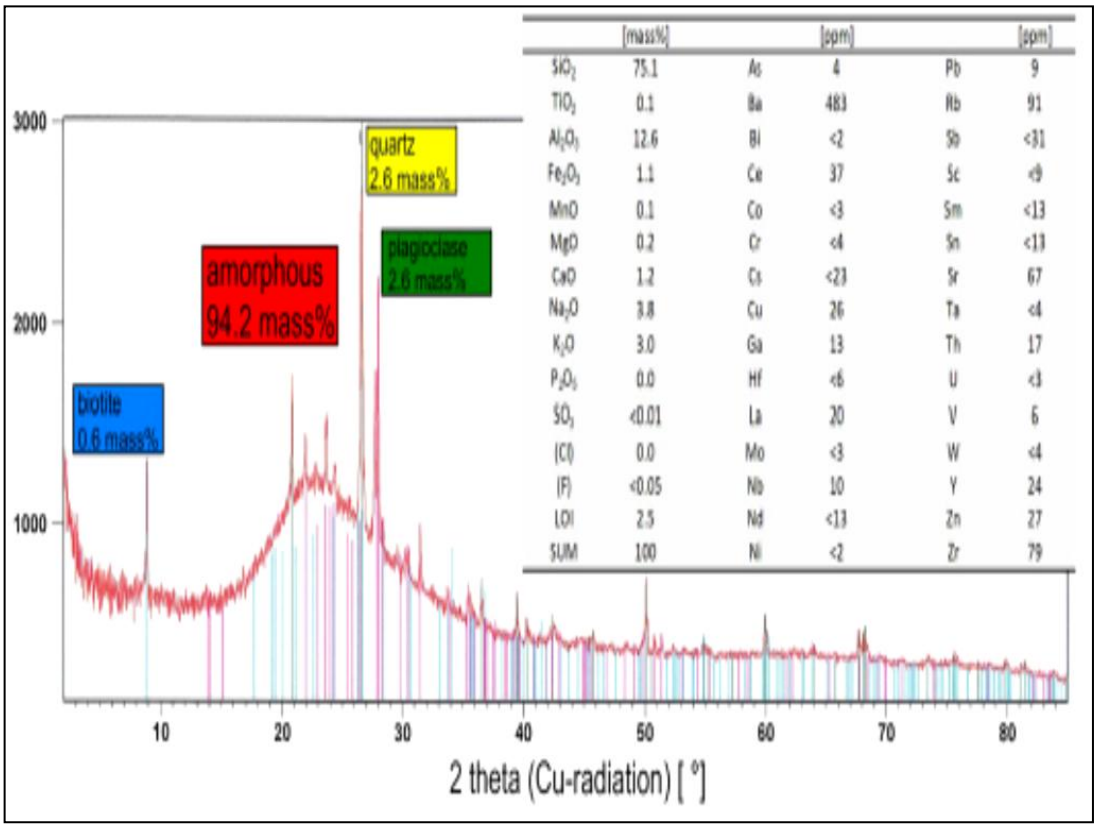


Figure 1.4. XRD patterns and chemical composition of Milos (Greece) perlite sample. (Source : Kaufhold, et al., 2014)

Expansion is made by using different types furnace. For example, a new fluidized sand-bed furnace was advanced and prepared expanded particles which have less than 15µm particle size and bulk density under 0.5 g/cm^3 (Sodeyama & Sakka, 2005). Also Sodeyama and Sakka were prepared lightweight alumina composites successfully using this expanded perlite which is named as Shirasuballons. Another study about furnaces, the exergoeconomy analysis of the two different perlite expansion furnace was used and compared the former and new perlite expansion furnaces (Gürtürk, Oztop, & Hepbaslı, Comparison of Exergoeconomic Analysis of Two Different Perlite, 2015). Also the energy efficiency was achieved better with exergoeconomy analysis which is an economic feasibility study (Gürtürk, Oztop, & Hepbaslı, Comparison of Exergoeconomic Analysis of Two Different Perlite, 2015). Other furnace is that the vertical electrical perlite expansion furnace which occurs the temperature control system (Angelopoulos, Gerogiorgis, & Paspaliaris, 2013). Other

one was used an entrained bed reactor and using the entrained bed reactor was observed the effects of residence time and riser temperature on the expanded perlite (Uemura, et al., 1999).

Water is one the factors that plays important role for expansion by reducing the viscosity and expanding the soft grain during evaporation (Zahringer, Martin, & Petit, 2001). After heat treatment, water desorption is a considerable feature for expansion because remaining water in the powder. It must contain enough water to expand in the powder until the softening point of the perlite (Sodeyama K. , Sakka, Kamino, & Tabata, Preparation and Characterization of Fine Shirasuballoons, 1996). FTIR spectra were used for Pitchstone and Obsidian to presence of water in the raw perlite (Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite, 1999). The band A is designated to the OH group of Si-OH, the band B is designated to water molecule vibration in the absorbed, the band C is from deformation vibration of water molecule. The band E is the Si-O stretching of the Si-OH group. After heating at 1000 ˚C the bands C and E vanished and the bands A and B to decrease. Figure 1.5.a. showed that FTIR spectra of the raw perlite which are Pitchstone and Obsidian and Figure 1.5.b. showed that after heat treatment at 1000 ˚C.

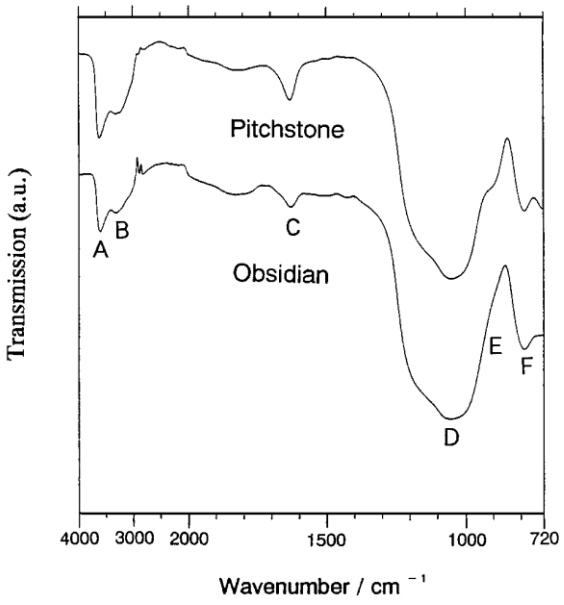


Figure 1.5.a. It was showed that FTIR spectra patterns for Obsidian and Pitchstone. (Source : Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite, 1999)

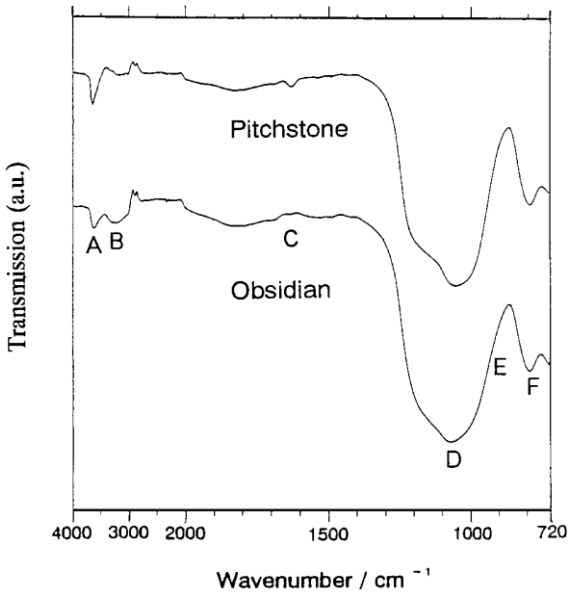


Figure 1.5.b. It was showed that FTIR spectra patterns after heating at 1000 ˚C for 1 min for Obsidian and Pitchstone.
(Source : Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite,1999)

In addition to literature, Roulia et all showed the effect of water content in perlite samples of different regions on expansion process (see Figure 1.6.). As a result this that the expansion ratio increase with total water content (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006).

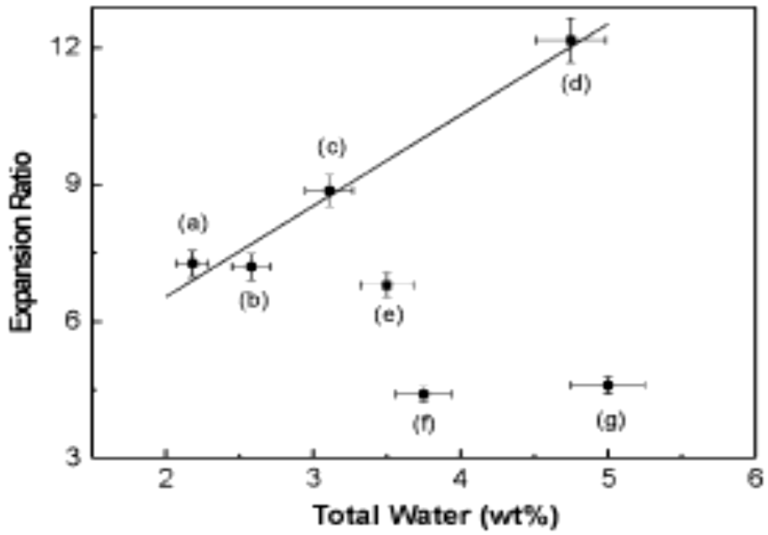


Figure 1.6. Effect of total water content on expansion ratio for perlite samples in different region (a) Provatas, (b) Tsigrado, (c) Trachilas, (d) Turkey, (e) Hungary, (f) Italy and (g) China.
(Source : Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006)

CHAPTER 2

EXPERIMENTAL

2.1. Materials

Samples of raw and expanded perlite were provided by the companies listed below. The origin of the raw perlite is also provided in the list (Table 3.1) and the code numbers of perlite samples for Bergama Mine in the list (Table 3.2). Images of the as-received and expanded samples from these sources are provided in Figures 2.1.a, 2.1.b, and 2.1.c.

Table 2.1. Source of raw perlite samples.

The Companies	**The Origins**
Çullas Group (Maltepe,İstanbul)	Aegean (Bergama Mine,İzmir)
Persan Group (Erzincan)	Eastern Anatolia (Mollatepe Mine,Erzincan)
Bülbüller Group (Kütahya)	Inner Western Anatolia (Avdan Mine,Kütahya)

Figure 2.1.a. Images of samples supplied from Bergama Mining Company in Maltepe İstanbul.

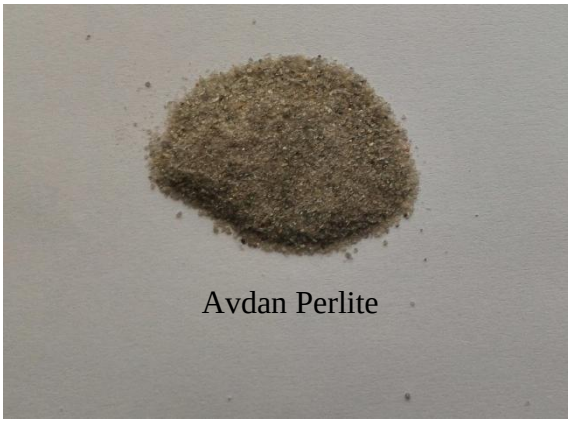


Figure 2.1.b. Image of sample supplied from Avdan Mining Company in Kütahya.

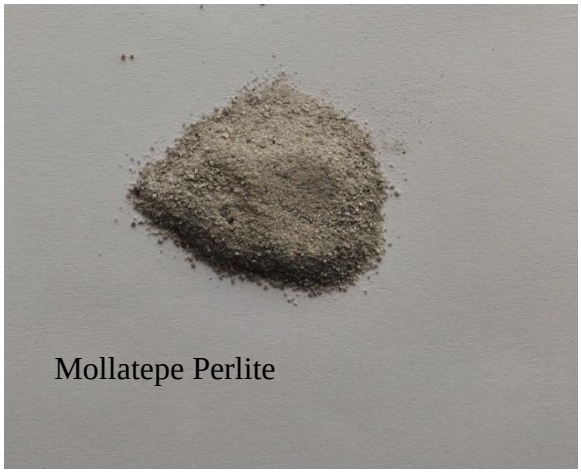


Figure 2.1.c. Image of sample supplied from Mollatepe Mining Company in Erzincan.

Taken from Çullas Group the code numbers of perlite were showed in the Table 2.2. Perlite samples are named according to the code numbers.

Table 2.2. The code numbers of perlite samples the region from Bergama Mine.

Code Numbers	**The Names Of Samples**
Bergama 01	Koyuneli Raw Perlite
Bergama 02	Köseler Raw Perlite
Bergama 03	Industrial Expansion

2.2. Processing of raw perlite

2.2.1. Sample Preparation

Samples of the as-received perlite were screened into six different grain size ranges using the following screen sizes: 500 μm, 400 μm, 315 μm -400 μm, 200 μm - 315 μm, 160 μm -200 μm, and -160 μm. The screens (Fritsch GmbH), were stacked, placing the smaller size at the bottom and larger size on the top. A bottom pan was placed to collect the fines passing the smallest screen size of 160 μm. The samples were screened for a total of 10 seconds. A total of 10g to 20g of perlite was screened for each sample. Sieving was done using Retsch- AS 200, Germany equipment.

2.2.2. Laboratory Heat Treatment and Expansion Experiments

Heat treatment experiments were conducted in an indirect electric heated front loading box type PLF/ PC442 1P-2S Series Protherm Furnaces, Ankara. Raw perlite samples were weighed and placed in a ceramic or platinum crucible (to be explained below) which were then positioned into the furnace. The sample crucibles were inserted in to the furnace at the targeted heat treatment temperature and were kept there for the targeted amount of heat treatment time. At the completion of the heat treatment time, samples were taken out of the furnace and were allowed to cool to room temperature.

Preliminary heat treatment experiments were performed using ceramic crucibles. The raw perlite samples were heat treated at 200˚C, 500˚C, 600 ˚C, 800˚C, 900˚C, 1000˚C, and 1200˚C temperatures for dwell times ranging between 5 minutes to 80 minutes. The expansion ability of the raw perlite as well as the experimental expansion conditions were optimized during these preliminary tests. Based on these findings expansion tests that are reported in this thesis were conducted in a 75ml platinum crucible at temperatures of 600˚C, 800˚C, 900˚C and 1000˚C. Samples in the platinum crucibles were placed into the oven at the targeted heat treatment temperature. Furnace was allowed to reach the target temperature after which each sample was kept in the furnace for 5 minutes after which they were extracted from the furnace and allowed to cool to room temperature.

The expansion behavior of the raw perlite samples were determined by comparing the bulk volume and bulk density of the material before and after the heat treatment procedure. Bulk density was measured by weighing the sample and placing this amount in a graduated cylinder to determine its bulk volume. The material was manually tapped for 3 minutes at approximately 60 taps per minute after which the bulk volume was read on the graduated cylinder. A comparison of the bulk volume of raw and expanded perlite in the graduated cylinder used for determining the bulk volume and density. Figure 2.2. shows that both of raw and expanded perlite samples with graduated cylinder.

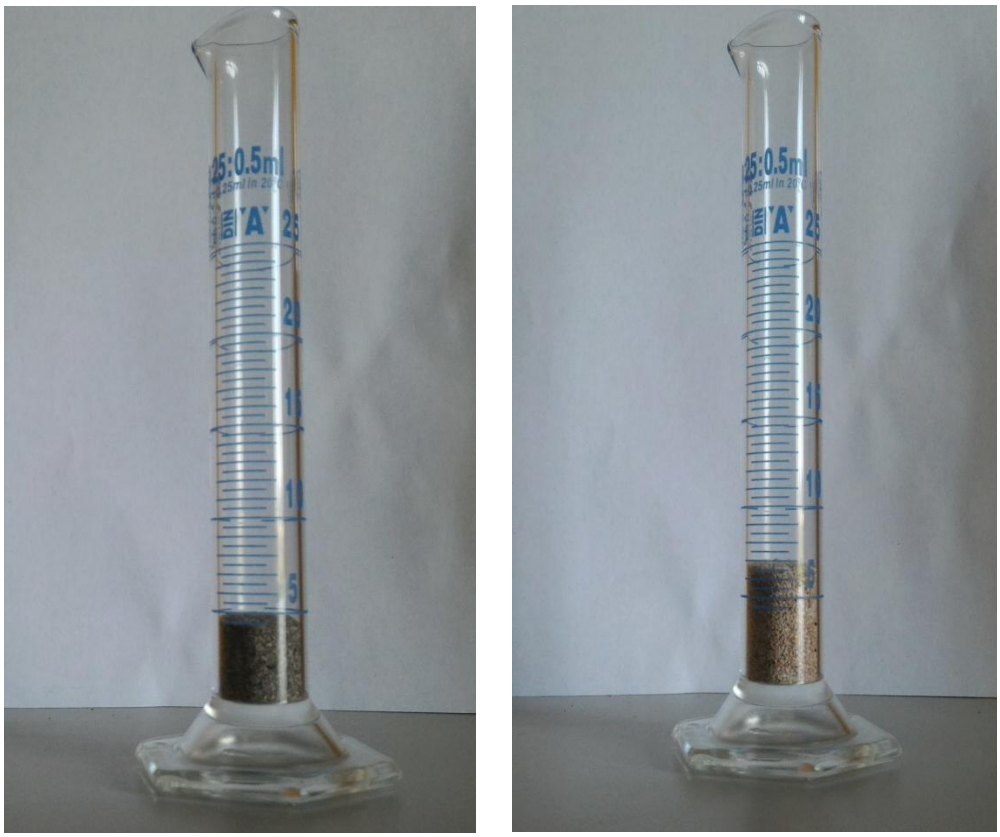


Figure 2.2. The raw and expanded perlite samples with graduated cylinder.

2.3. Materials Characterization

X-ray diffraction (XRD), thermogravimetric analysis (TGA), X-ray fluorescence (XRF), scanning electron microscopy (SEM), optical microscopy, and particle size distribution techniques were used for the characterization of perlite samples. Below sections provide a detailed explanation of the equipment and procedure used in the corresponding analysis technique.

2.3.1. X-Ray Diffraction (XRD) Analysis

XRD was used to observe the presence amorphous and crystal in the perlite. The analysis was performed in the Bragg-Brentano focusing geometry from 5˚ to 80˚, with a scan step size set at 0.139 ˚/minute and radiation at 45 kV, 40 mA on Phillips X'Pert Pro MPSS Powder X-Ray diffraction equipment. The collected patterns were analyzed for the identification of any crystalline constituents using search match software (Highscore Plus). Amount of crystalline phase was also determined from the calculation and output provided by the software.

2.3.2. Thermogravimetric (TGA) Analysis

The (Thermal Gravimetric Analysis) TGA examines the change in mass of a material as a function of temperature. The analysis on the perlite samples were conducted in between 30˚C to 800˚C at a heating rate of 10 ˚C/min under nitrogen purge. The measurements were performed using Perkin Elmer Diamond TGA/DTA equipment.

2.3.3. X-Ray Fluorescence (XRF) Analysis

Chemical composition of the perlite was determined both oxide state and elemental constituents of the material using XRF. The measurements were performed using a Spectro IQ-II instrument. Perlite was prepared into a glass disk using lithium tetraborate as the flux material.

2.3.4. SEM Analysis

Raw and expanded perlite morphologies were examined using scanning electron microscopy (SEM) together with energy dispersive x-ray (EDX) for a qualitative elemental analysis were employed on carbon tape. Analyses were conducted using a Quanta 250 FEG model and Philips XL 30S FEG model SEM's.

2.3.5. Optical Microscopy Analysis

Optical microscopy was used to taken images of the raw perlite samples and after heat treatment the expanded perlite in different regions using Olympus BX53 in German, Japan and Binocular Microscope-Olympus CX31 with camera DP-25 in Japan.

CHAPTER 3

RESULTS AND DISCUSSIONS

3.1. Chemical Analysis of Perlite

The importance of the chemical make-up, especially the presence and amount of crystalline phase in raw perlite was discussed in section 1. Results of the chemical composition, as measured using x-ray fluorescence (XRF) spectroscopy, and crystalline/amorphous nature, as measured by x-ray diffraction (XRD) of both the raw and expanded perlite are given in this section. These results will form the basis for defining the variations in the expansion behavior of the different sources of perlite examined in this study.

3.1.1. X-Ray Diffraction Analysis Results

Presence of amorphous and crystalline phase of perlite was evaluated using X-ray diffraction (XRD). The XRD measurement protocol is explained in section 2.3. Only the as-received raw perlite samples were evaluated for the phase analysis.

XRD patterns of raw perlite samples from Bergama (İzmir) region (01 and Bergama 02), Avdan (Kütahya) region and Mollatepe (Erzincan) region are provided in Figures 3.1.a. - 3.1.b. - 3.1.c. and 3.1.d., respectively.

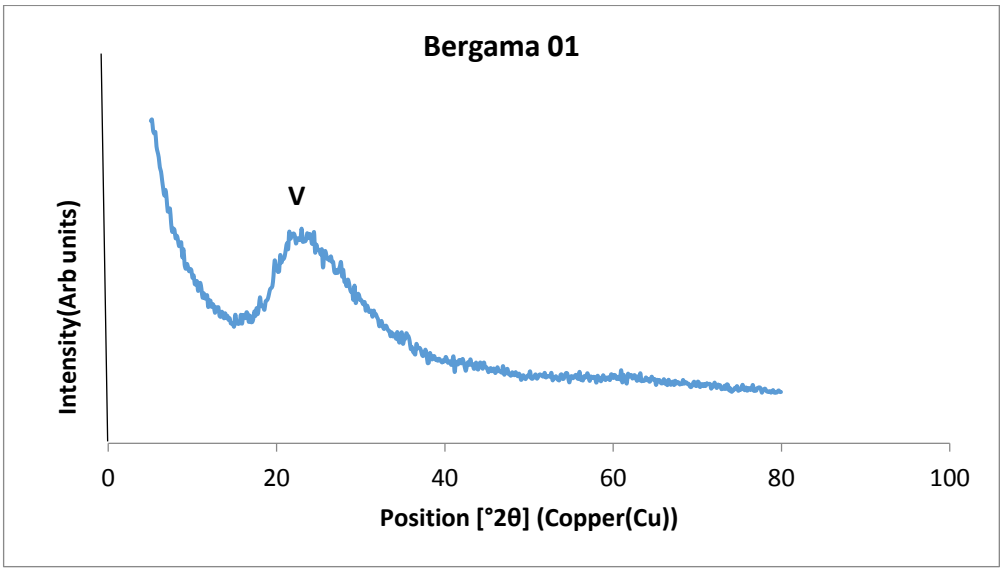


Figure 3.1.a. XRD Pattern of Bergama Mining (raw perlite sample) in İzmir (V: Vermiculite).

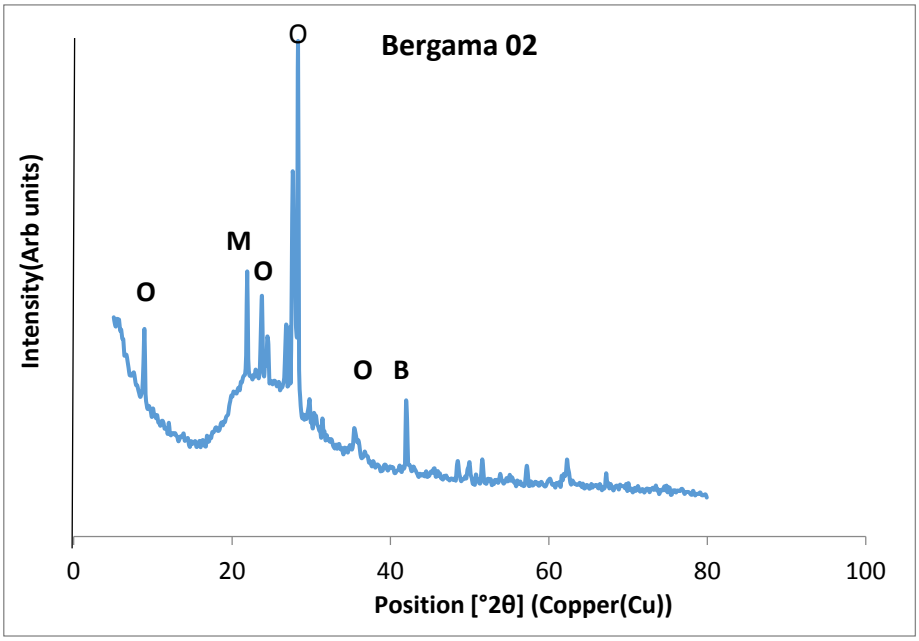


Figure 3.1.b. XRD Pattern of Bergama Mining (raw perlite sample) in İzmir (O: Oligoclase), (M: Montmorillonite), (B: Boggsite).

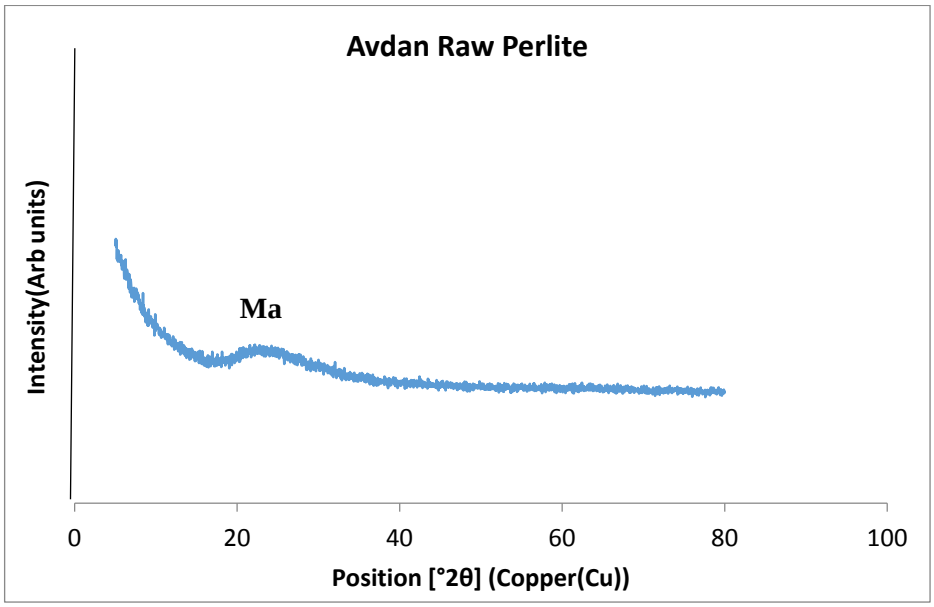


Figure 3.1.c. XRD Pattern of Avdan Mining (raw perlite sample) in Kütahya (Ma: Mayenite).

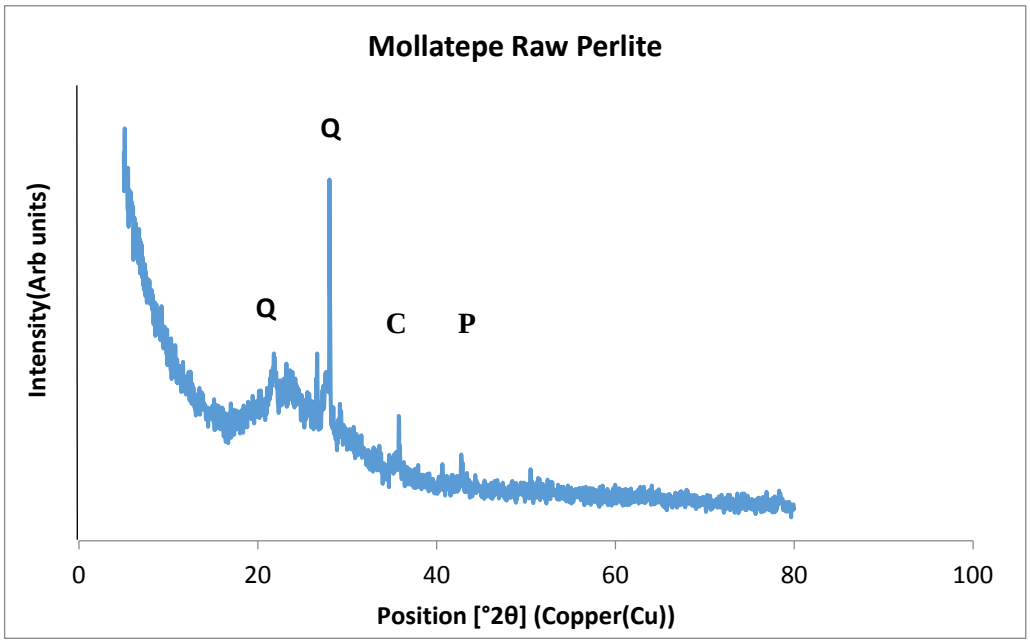


Figure 3.1.d. XRD Pattern of Mollatepe Mining (raw perlite sample) in Erzincan (Q: Quartz), (C: Cristobalite), (P: Periclase).

Figures 3.1.a to 3.1.d. show that the raw perlite samples were mostly amorphous with presence of crystalline phase found for samples from Bergama (02) and Mollatepe. Table 3.1. provides the crystal compounds identified from the XRD pattern analysis. The data suggest that the four samples examined in this study possess different crystalline compounds. Samples obtained from Avdan indicate that mayenite was the only crystalline compound present while the Mollatepe sample showed presence of quartz, cristobalite and periclase. Sample Bergama-01 shows presence of vermiculate while Bergama-02 shows a combination of the presence of oligoclase, montmorillonite and boggsite. This difference from the same region of perlite illustrates how the character of perlite can vary when extracted from the same mine. It is highly likely that these two samples were extracted from different flow lines of the lava when perlite formation took place. It is also likely that different soil layer formations, example montmorillonite, may have entered the material as a contamination. In a similar manner, samples from Avdan and Mollatepe show presence of different crystal formations.

Table 3.1. Crystal compounds and their chemical formulas identified by XRD analysis of raw and expanded perlite samples.

Compound Name	**Chemical Formula**	**Sample Where Compound is Identified**
Vermiculite (V)	$(Mg,Fe)_3[(Al,Si)_4O_{10}](OH)_2 \cdot 4H_2O$	Bergama 01
Oligoclase (O)	$(Ca,Na)(Al,Si)_4O_8$	Bergama 02
Montmorillonite(M)	$Al_4Si_8O_{24}$ Ca	Bergama 02
Boggsite(B)	$NaCa_2(Al_5Si_{19}O_{48}) \cdot 17(H_2O)$	Bergama 02
Mayenite(Ma)	$Ca_{24}Al_{28}O_{64}$	Avdan
Quartz(Q)	Si_3O_6	Mollatepe
Cristobalite(C)	Si_4O_8	Mollatepe
Periclase(P)	Mg_4O_4	Mollatepe

Table 3.2. shows the results of the amounts of crystalline phase determined from raw perlite samples based on the analysis of the search match software. The results show that the crystalline amounts of the four samples varied between 1.75 to 10.19 %, with the highest value measured from Bergama-02 and the lowest from Bergama-01. While it can be argued if these results are statistically significantly different from each other, it provides a grounds for a comparison between the lowest and highest values measured. This is important because the presence and extent of a crystalline phase is known the effect the expansion behavior of perlite during a heat treatment process (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006). This will further be discussed in sections 3.2. and 3.3. of this chapter.

Table 3.2. Results of crystalline amounts in raw perlite samples.

Sample Source (Raw Perlite)	**% Crystalline**
Bergama 01	1.75%
Bergama 02	10.19%
Avdan	3.08%
Mollatepe	5.02%

Overall, the XRD analysis of the samples examined in this study compares well with the findings provided in the literature. Perlite has been determined to predominantly possess an amorphous structure together with presence of certain amount of crystalline phases (see Figure 3.7.) (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006), (Ciullo, 1996). Sodeyama (Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite, 1999) has shown the crystalline amount to be about 95 weight % for perlite samples analyzed from Tensin, China and Padang, Indonesia. Roulia (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006) further shows that these small crystallites in raw perlite prevent the expansion of the grain during a heat treatment process.

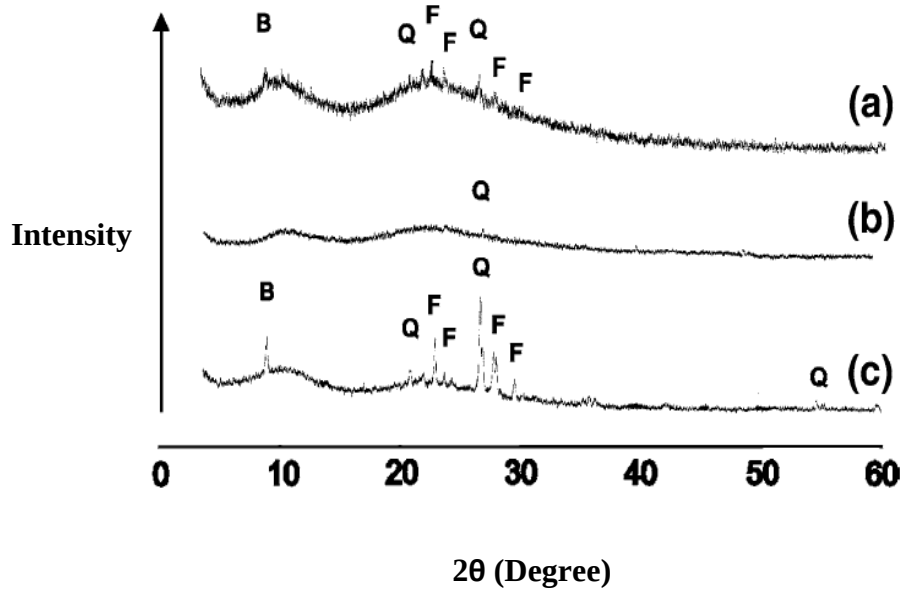


Figure 3.7. XRD patterns of perlite samples from Trachilas region (a) medium grain in the raw perlite, (b) expanded perlite, and (c) unexpanded states with B (biotite), F (feldspar), and Q(quartz).
(Source : Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006).

3.1.2. X-Ray Fluorescence Analysis Results

Table 3.4. shows the XRF chemical composition results for the four sources of raw perlite examined in this study and typical chemical composition of perlite (Perlite Institute, 2011). Results show the majority (above 90 weight %) of the composition to be made up of SiO_2, Al_2O_3, K_2O, Na_2O, Fe_2O_3, and CaO. The composition analysis shows presence of minor elements, which are all below 1%, to be made up of a mixture of MgO, TiO_2, MnO, BaO, Ta_2O_5, CuO. XRD results given and discussed in section 3.1.1. for these samples had shown the raw forms of perlite to contain a certain amount of a crystalline phase. This amount was determined to be less than 10%. The XRF results together with XRD findings, therefore, suggest that the raw perlite samples are predominantly glass and that this glass is of an alkali-alkali earth-alumino-silicate based chemistry. The crystalline compounds shown in Table 3.1. also match well with the ingredients found in the XRF analysis results.

Table 3.4. X-ray Fluorescence Spectrometer (XRF) results for 01 and Bergama 02 from Bergama Mine, Avdan Mine, and Mollatepe Mine. Results are given in weight %.

Chemical Composition	**Bergama 01**	**Bergama 02**	**Avdan**	**Mollatepe**	**Typical Chemical composition of perlite**
SiO_2	72.80%	71.25%	73.40%	70.00%	71.00-75.00%
Al_2O_3	13.10%	14.50%	15.90%	15.70%	12.50-18.00%
K_2O	4.30%	4.20%	5.40%	5.00%	4.00-5.00%
Na_2O	2.10%	2.04%	1.80%	5.50%	2.90-4.00%
CaO	1.00%	1.15%	1.00%	1.10%	0.50-2.00%
Fe_2O_3	1.00%	1.25%	1.00%	1.70%	0.10-1.50%
MgO	0.10%	0.20%	0.30%	0.40%	0.03-0.50%
TiO_2	0.20%	0.30%	-	-	0.03-0.20%
MnO	0.01%	0.07%	0.10%	0.01%	0.00-0.10%
BaO	-	-	0.30%	-	0.00-0.10%
Ta_2O_5	-	-	1.00%	1.00%	0.00-0.10%
CuO	-	0.60%	-	-	0.00-0.10%
Total	94.61%	95.56%	100.20%	100.41%	

3.2. Expansion of Raw Perlite

It is known that the expansion of raw perlite takes place with the removal of water (Varuzhanyan, Varuzhanyan, & Varuzhanyan, 2006). Accordingly, this section provides the results and findings for the expansion of raw perlite based on the amount and release of water as a function of temperature from its structure. Results from thermo gravimetric analysis (TGA) of raw perlite if first presented followed by the results and analysis obtained in the amount of expansion measured when raw samples have been heat treated at different temperatures. Findings on the morphology of raw and expanded perlite samples are also provided based on scanning electron microscope (SEM) and optical microscope (OM) examinations.

3.2.1. Thermal Gravimetric Analysis (TGA) Results

TGA results for the four raw perlite samples examined in this study are shown in Figures 3.8. to 3.11. The measurement conditions have been provided in section 2.3.2. TGA results illustrate the reduction in weight, which is predominantly attributed to the loss of water, as a function of temperature is shown in the figures. The onset ($T_{initial}$) and completion (T_{final}) of the weight loss temperatures are also provided in each figure. These temperatures have been determined from where the slope of the TGA curve becomes zero. These results are tabulated and presented in Table 3.5. Data suggests that the water content of the Bergama samples are high when compared with samples from Mollatepe and Avdan. When the onset and completion temperatures of water release are compared, Mollatepe sample appears to show a difference compared with the other three samples: Mollatepe samples show onset temperature at 190 ˚C compared with 247-264 ˚C temperature range for the other three; in a similar manner, the completion of water release for Mollatepe appears at 650 ˚C compared with 696-681 ˚C temperature range for the other three.

The total amount of water present in the four perlite samples appear to be in a good agreement with the results reported in the literature which states the water amounts to be between 1.5-5.0 weight % (Sodeyama & Sakka, 2005), (Jing, Fang, Liu, & Liu, 2011). The TGA results also shows the temperatures and its range where water

is released to occur over a wide range, covering the evaporation of the different forms of water present in the samples. Both the total amount of water and the temperatures and the ranges of temperatures of the release of water relates to the ability of perlite to expand. This will be further discussed in relation to the results from TGA measurements in Section 3.3. of this chapter.

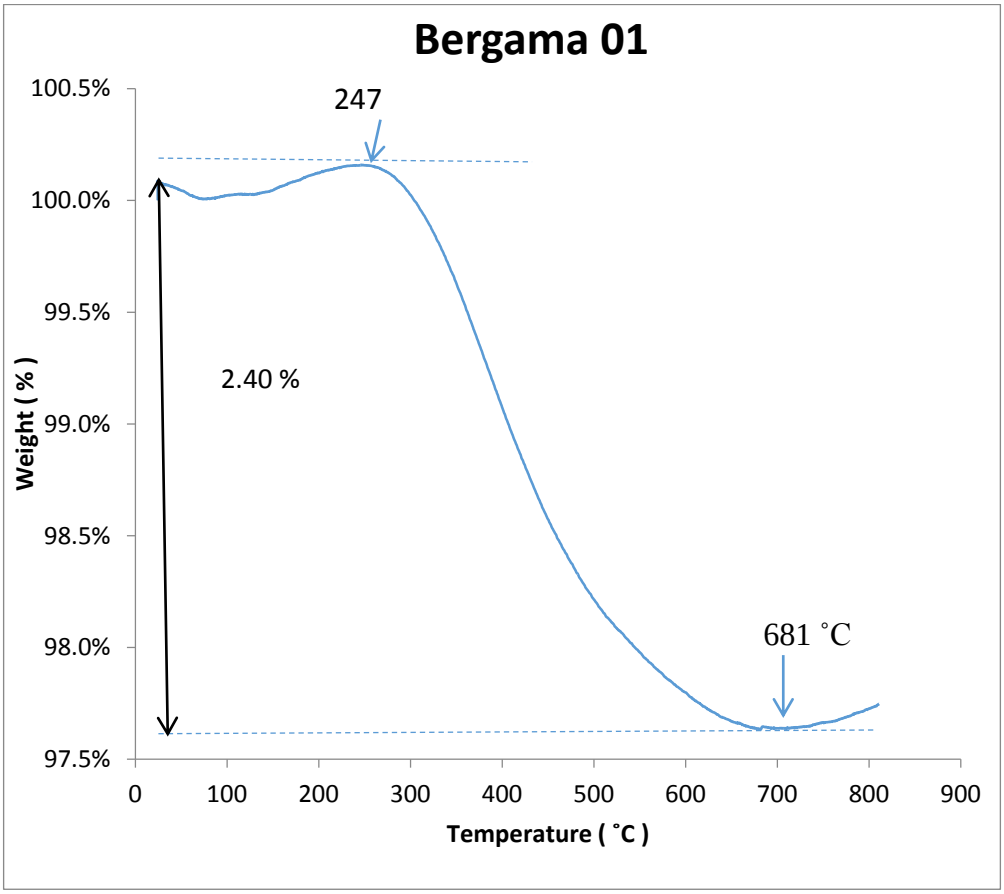


Figure 3.8. TGA results of raw perlite sample (Bergama 01) from Bergama region.

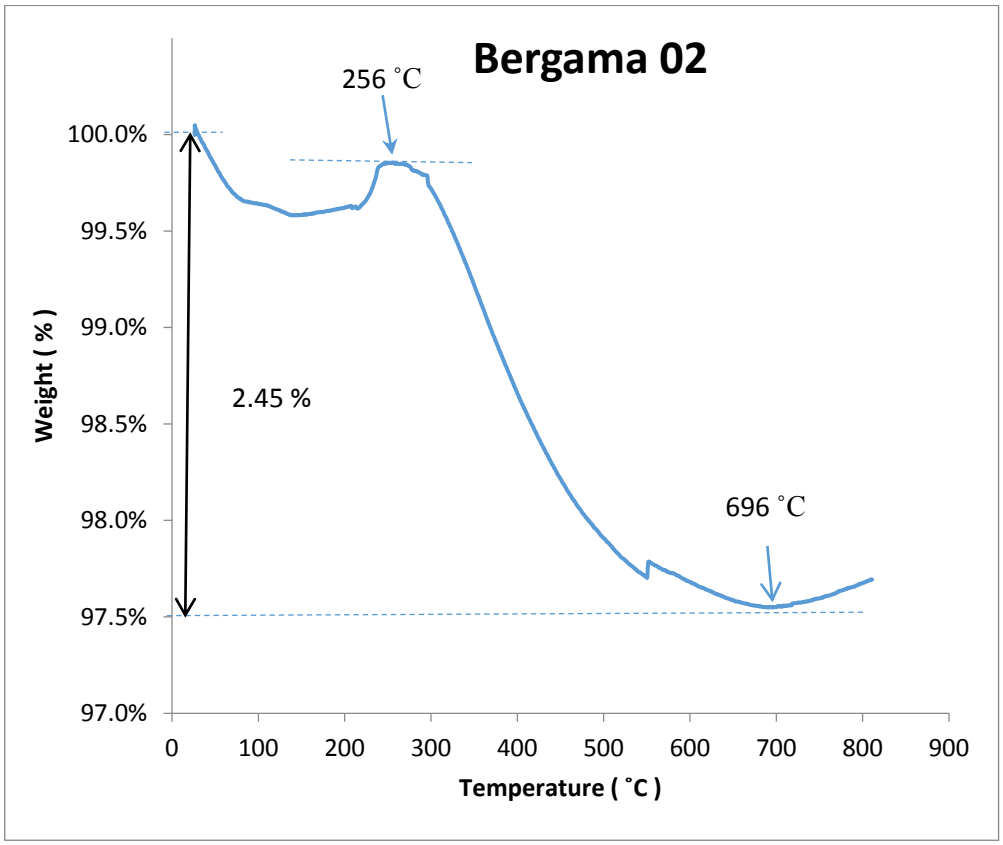


Figure 3.9. TGA results of raw perlite sample (Bergama 02) from Bergama region.

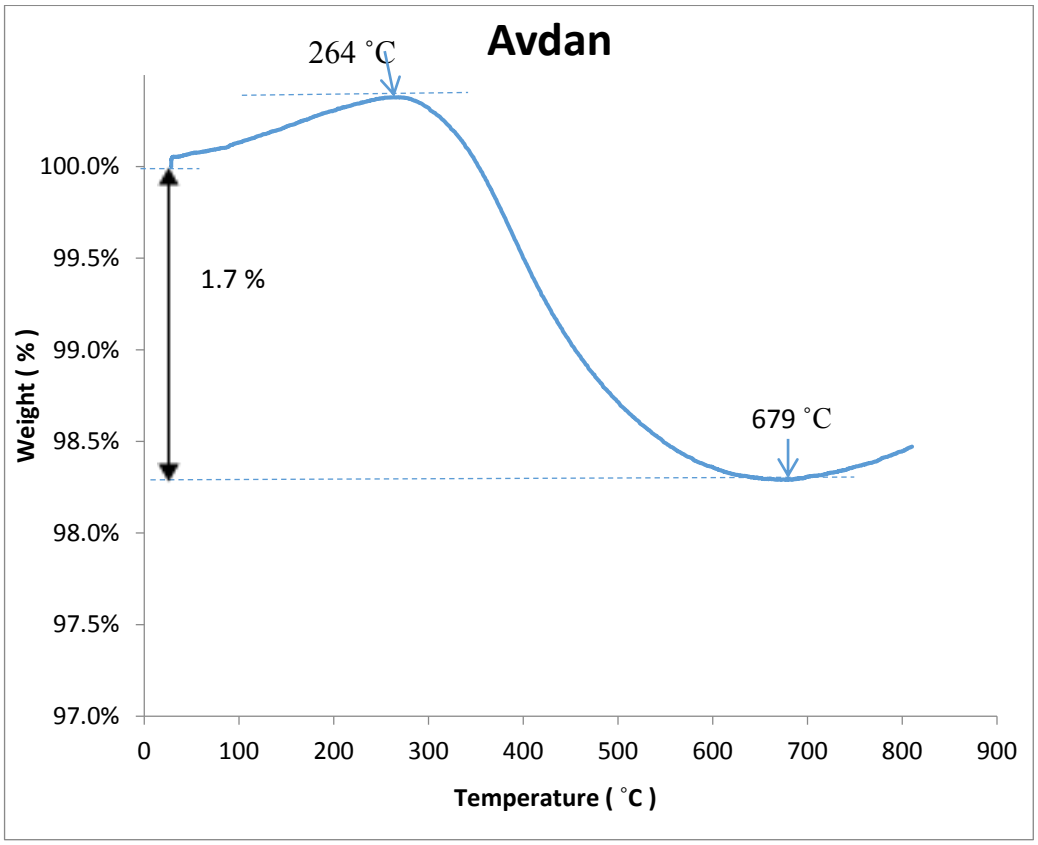


Figure 3.10. TGA results of raw perlite from Avdan.

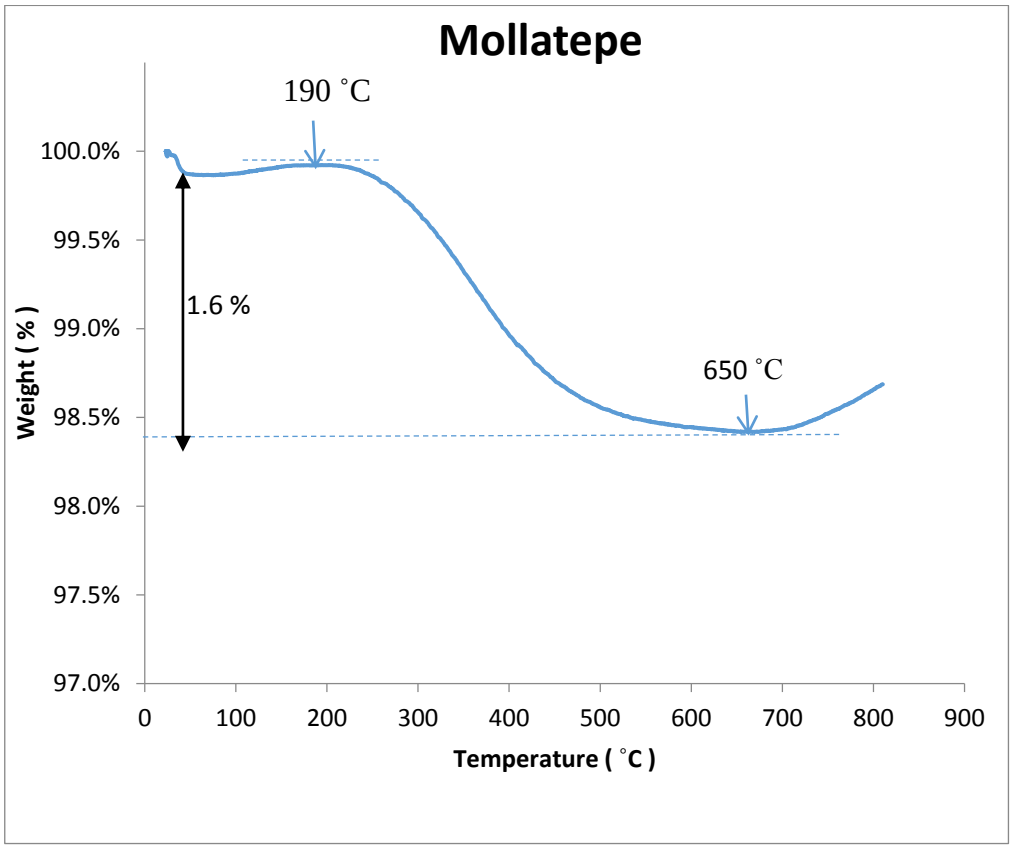


Figure 3.11. TGA results of raw perlite from Mollatepe.

Table 3.5. Summary of total reduction in weight, the onset (Tinitial) and end (Tfinal) of the weight loss data determined from TGA analysis for each raw perlite sample.

Samples of Region	**Loss of weight %**	**$T_{initial}$**	**T_{final}**
Bergama 01	2.40 %	247 ˚C	681˚C
Bergama 02	2.45%	256 ˚C	696 ˚C
Avdan	1.7 %	264 ˚C	679 ˚C
Mollatepe	1.6%	190 ˚C	650 ˚C

3.2.2. Laboratory Expansion Results

The expansion of perlite was determined by comparing the initial and final bulk volume of raw and expanded perlite after heat treatment, as explained in section 2.2. Table 3.6 shows the results of the percentage of volume increase when the raw perlite was heat treated at 600 ˚C, 800 ˚C, 900˚C and 1000 ˚C. Figure 3.12.a-d. shows the results given in Table 3.6 in a graphical format. The expansion was also calculated based on density change, the data and figures of which are provided in the Appendix. The results suggest that all samples show an increase in volume expansion with increased heat treatment temperature. The expansion has been found to be negligible below 600 ˚C. Four samples show a difference in their expansion volume, with Bergama 01 sample showing the highest expansion and Mollatepe and Bergama 02 samples showing the least amount. The effect of particle size is also significant with expansion amount decreasing as particle size decreases. This effect becomes more significant as the heat treatment temperature increases. Sample from Avdan also

appeared to show a difference in its expansion behavior where its expansion was very low at 600 ˚C, increasing at 800 ˚C but at lower amount compared to Bergama. The expansion of Avdan perlite appeared to increase after 800 ˚C.

It is important to note here that the expansion results collected in this study represents a comparative evaluation of the ability for the different samples to show an expansion capacity. The results reflect the change in volume and density of raw perlite when heat treated at the target temperatures for a period of time that is measured in minutes. This expansion compares with the treatment times measured in few seconds for expanded perlite processed at industrial scale furnaces. Hence, the findings in this study may not completely match the bulk volumes and densities that are obtained from commercial products but the relative trends in the volume and density change measured in this study will help provide an understanding on the capacity of the raw material to expand. These results will be further discussed by combining it with the findings from the other measurements of this study in section 3.3 of this chapter.

In addition to, in this study the expansion process was carried out temperatures between at 600-1000 ˚C. The results given here is valid up to 1000 ˚C. It could not measured expansion in temperature over 1000 ˚C due to forming adhesion between each other particles in this material (more details in section 3.3.).

Table 3.6. Volume % expansion data for the four perlite samples measured at 600 ˚C, 800 ˚C, 900˚C and 1000 ˚C. The results are given for four different size ranges of raw perlite before expansion.

Perlite Source	Heat Treatment Temperature	Particle Size Range (µm)			
		400-500 µm	315-400 µm	200-315 µm	160-200 µm
Bergama 01	600˚C	14%	13%	6%	6%
	800˚C	87%	52%	42%	12%
	900 ˚C	121%	89%	63%	61%
	1000˚C	202%	198%	138%	132%
Bergama 02	600˚C	12%	5%	5%	4%
	800˚C	28%	43%	17%	16%
	900 ˚C	66%	51%	28%	18%
	1000˚C	67%	85%	31%	28%
Avdan	600˚C	1%	2%	2%	0%
	800˚C	45%	35%	27%	35%
	900 ˚C	93%	102%	105%	83%
	1000˚C	159%	135%	151%	136%
Mollatepe	600 ˚C	0%	3%	1%	1%
	800˚C	40%	28%	26%	27%
	900 ˚C	42%	30%	28%	32%
	1000˚C	40%	28%	26%	27%

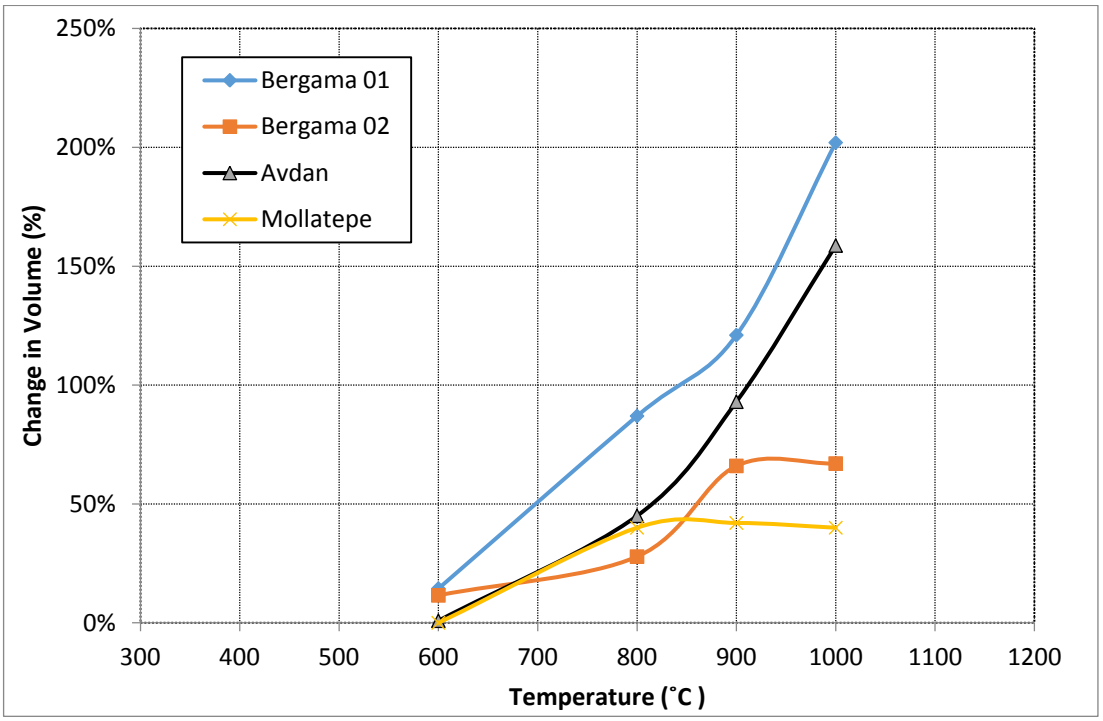


Figure 3.12.a.The percentage of increasing in volume with increasing temperature all of the perlite samples at 400-500µm.

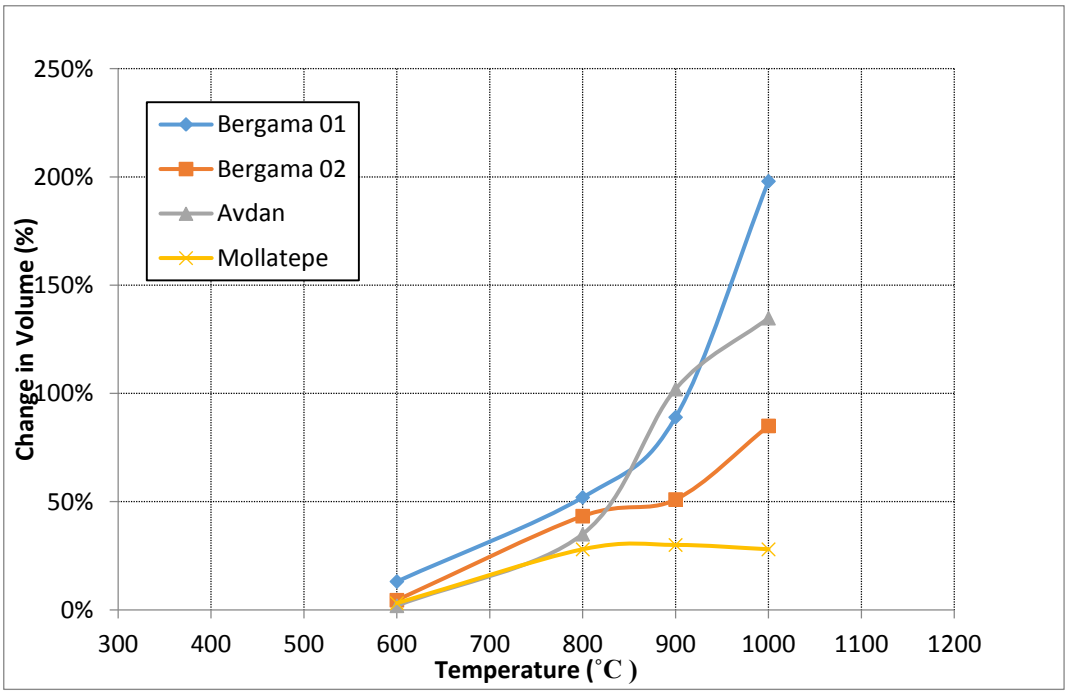


Figure 3.12.b. The percentage of increasing in volume with increasing temperature all of the perlite samples at 315-400µm.

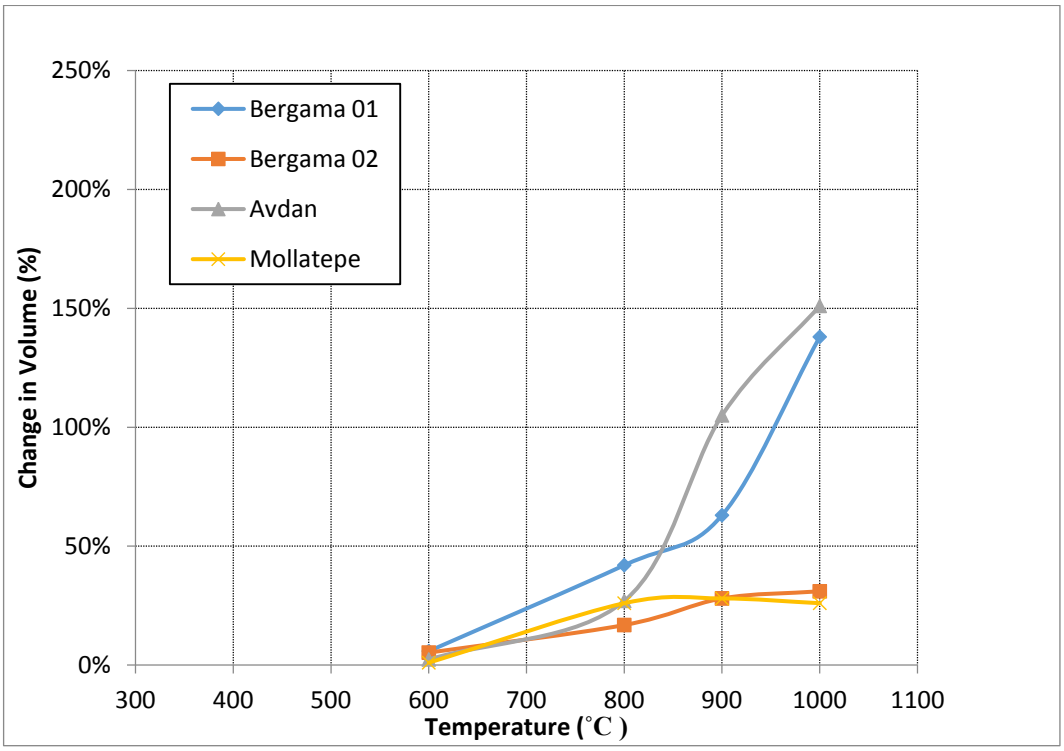


Figure 3.12.c. The percentage of increasing in volume with increasing temperature all of the perlite samples at 200-315µm.

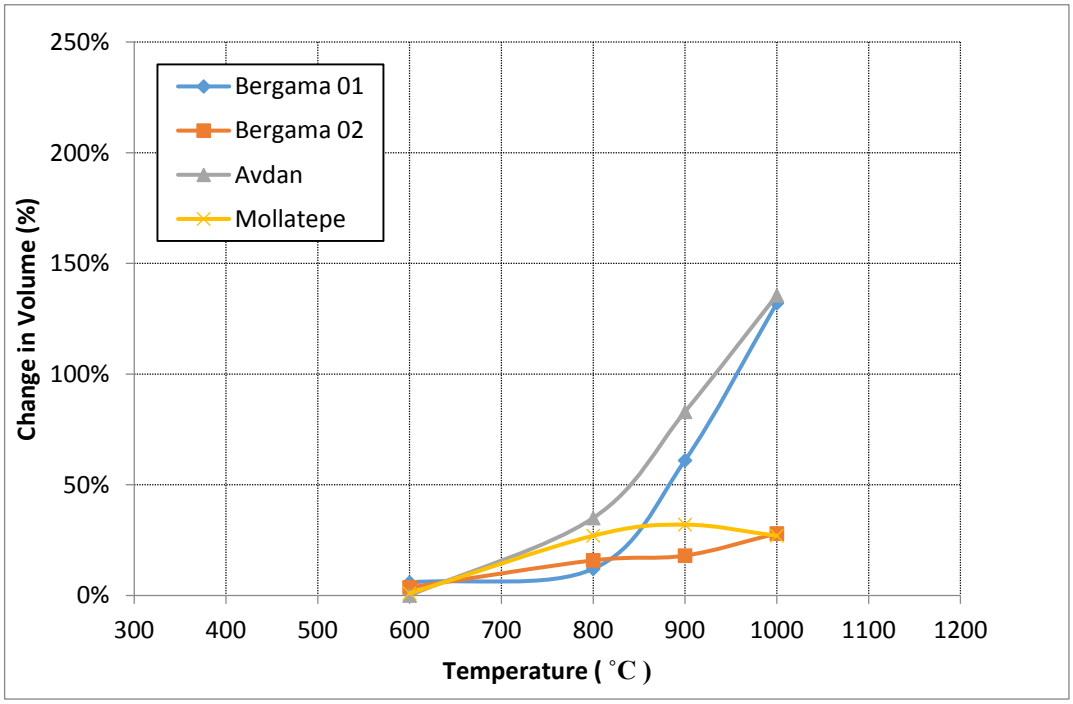
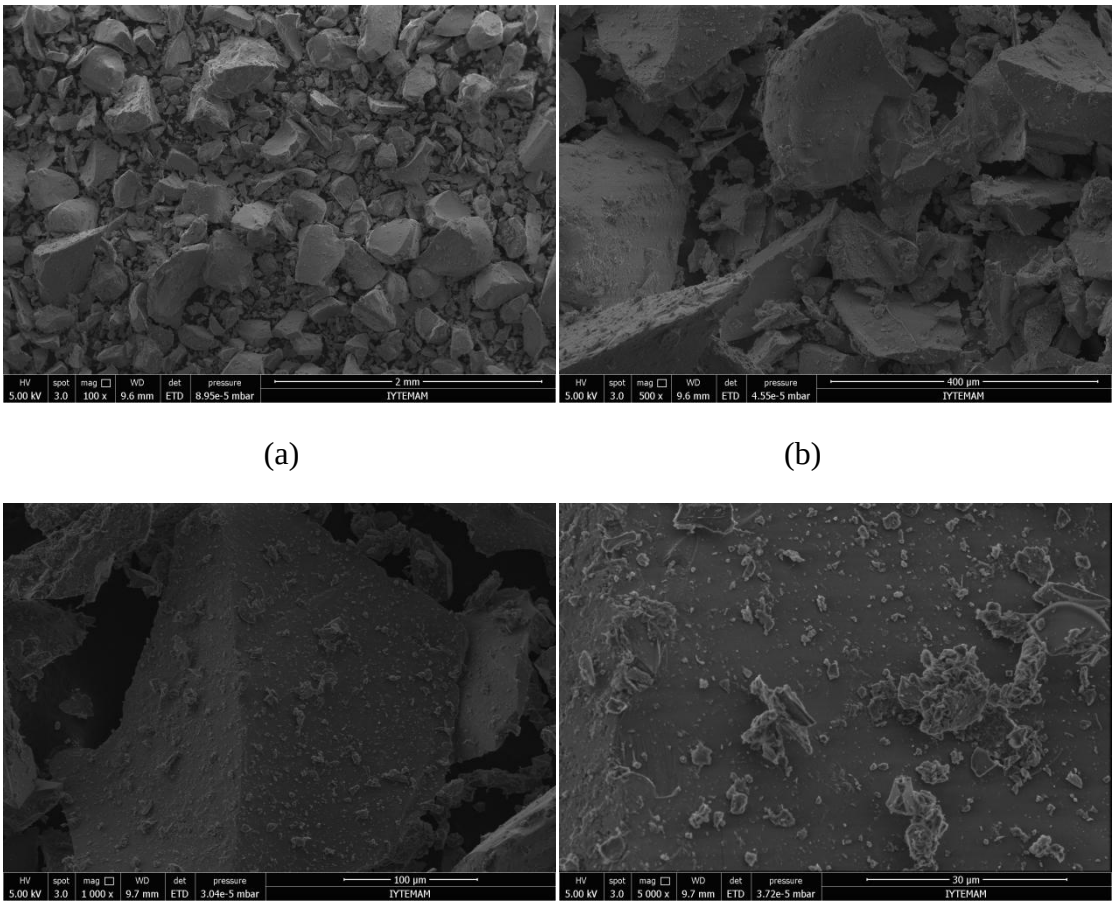


Figure 3.12.d. The percentage of increasing in volume with increasing temperature all of the perlite samples at 160-200µm.

3.2.3. Morphology and Microscopy Results

SEM was used to determine to grain morphology of raw and expanded perlite samples and to observe changes of raw perlite samples after heat treatment. Also, it was used to take images of raw and expanded perlite samples using Optical microscopy. It was carried out the most available magnifications, sizes and temperatures all of the perlite samples both SEM and Optical microscopy.

Figures 3.13.a-d. showed the SEM micrographs of raw perlite samples from Bergama 01, Bergama 02, Avdan and Mollatepe, respectively. As seen like that in each the raw perlite samples definition, there are particles at variant sizes, which are properly cracked, and have smooth surface. In here, there is one noteworthy difference (see figure in 3.13.d.), in Mollatepe perlite samples, particles, with more rough surface with layers than other's surface, are seen. This property is not present by other the three samples.



(a) (b)

(c) (d)

Figure 3.13.a. Raw perlite SEM images from Bergama 01 samples taken at different magnifications: (a) 100x, (b) 500x, (c) 1000x, (d) 5000x.

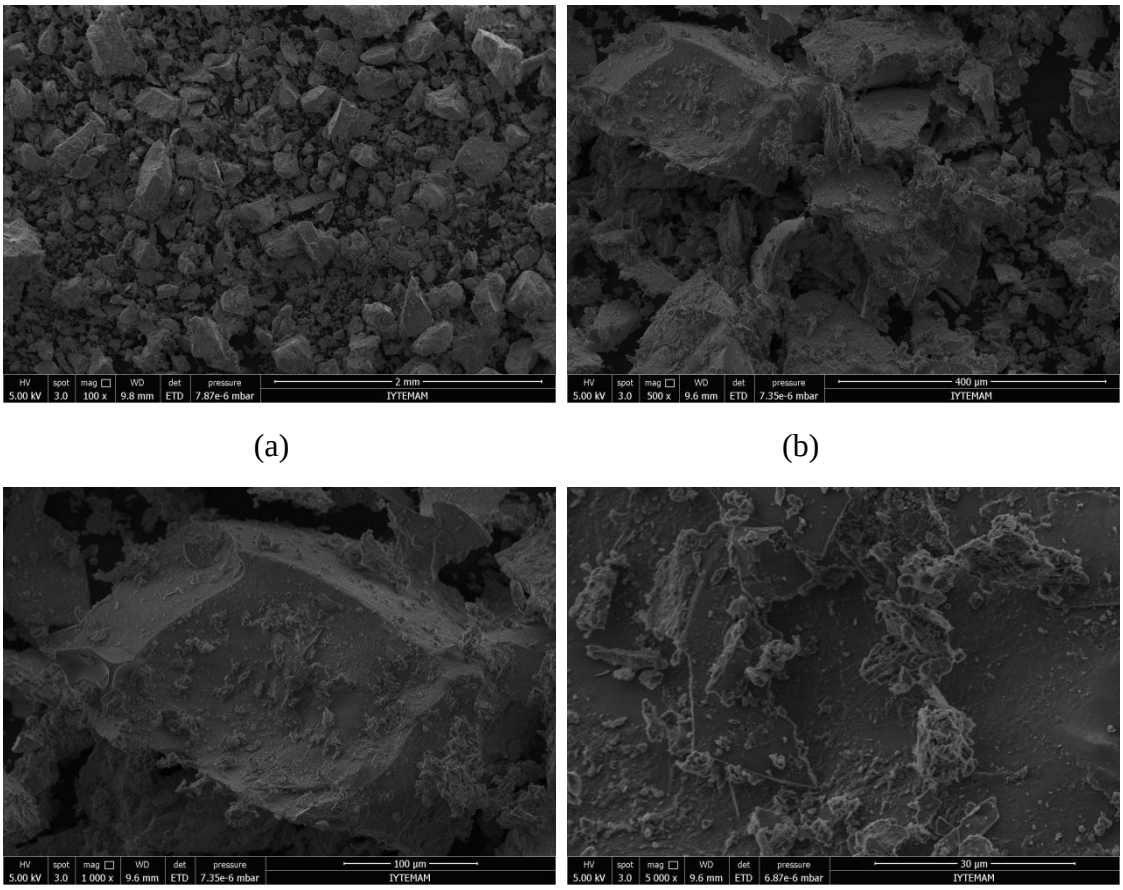
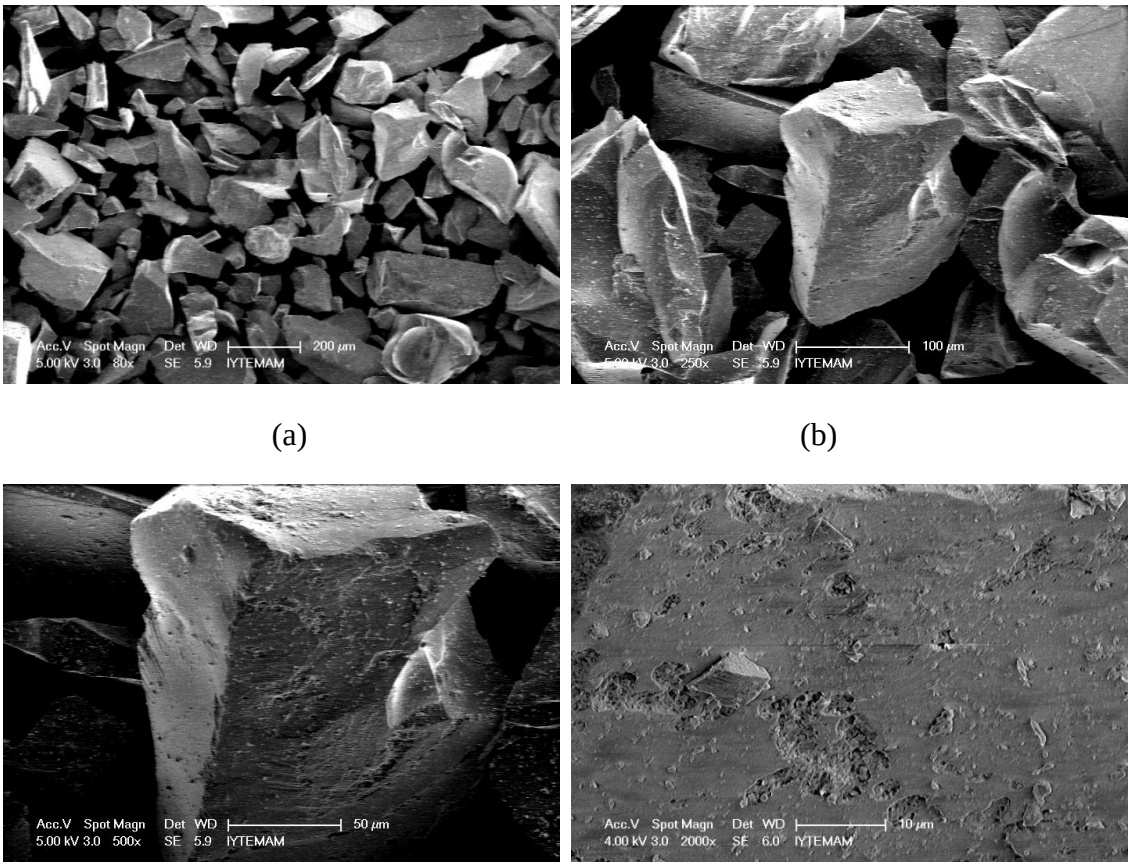


Figure 3.13.b. Raw perlite SEM images from Bergama 02 samples taken at different magnifications: (a) 100x, (b) 500x, (c) 1000x, (d) 5000x.



(a) (b)

(c) (d)

Figure 3.13.c. Raw perlite SEM images from Avdan samples taken at different magnifications: (a) 80x, (b) 250x, (c) 500x, (d) 2000x.

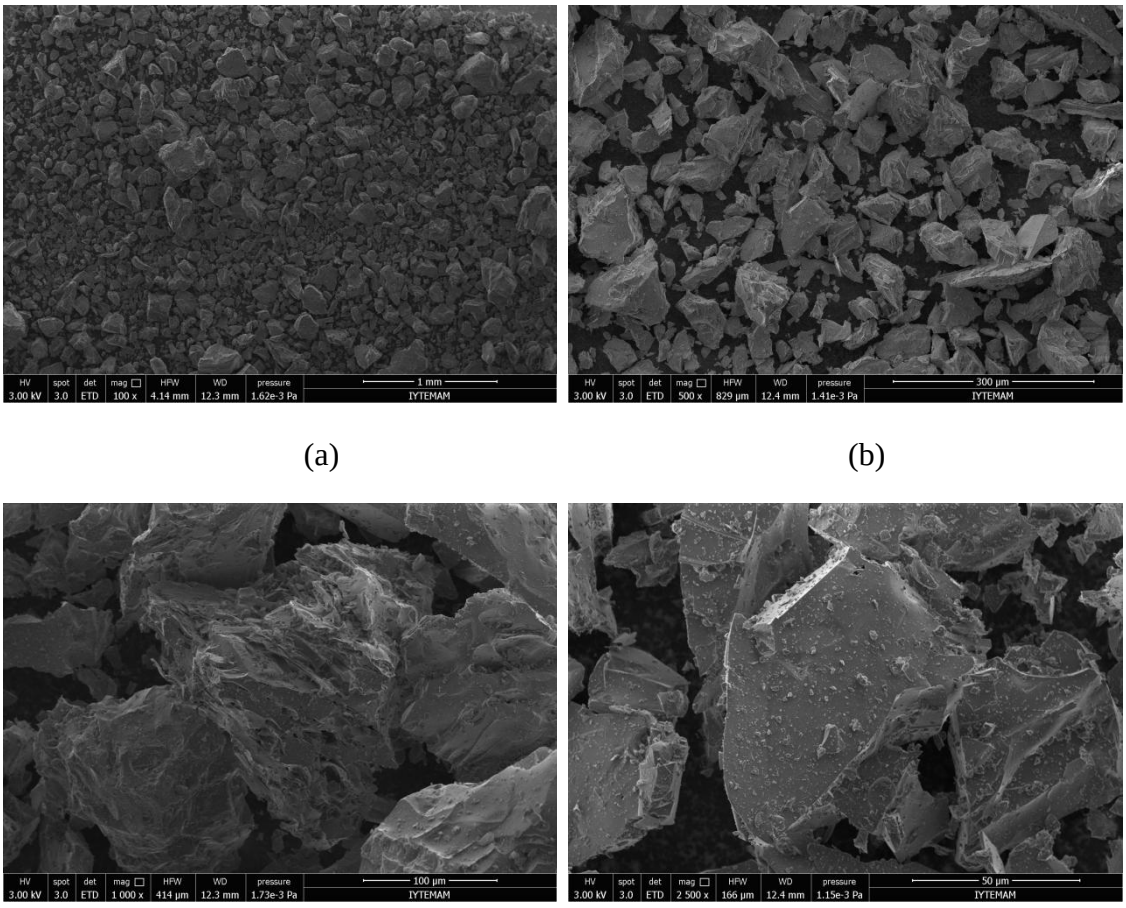
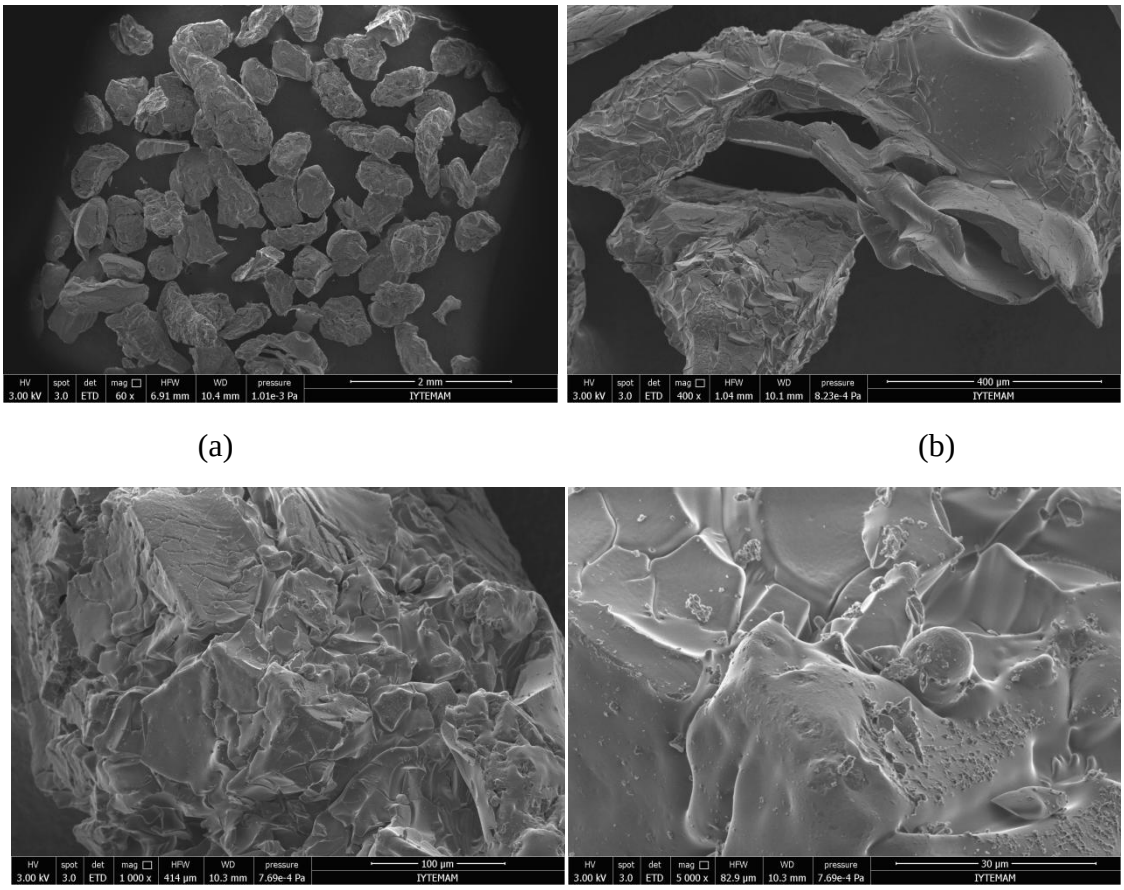


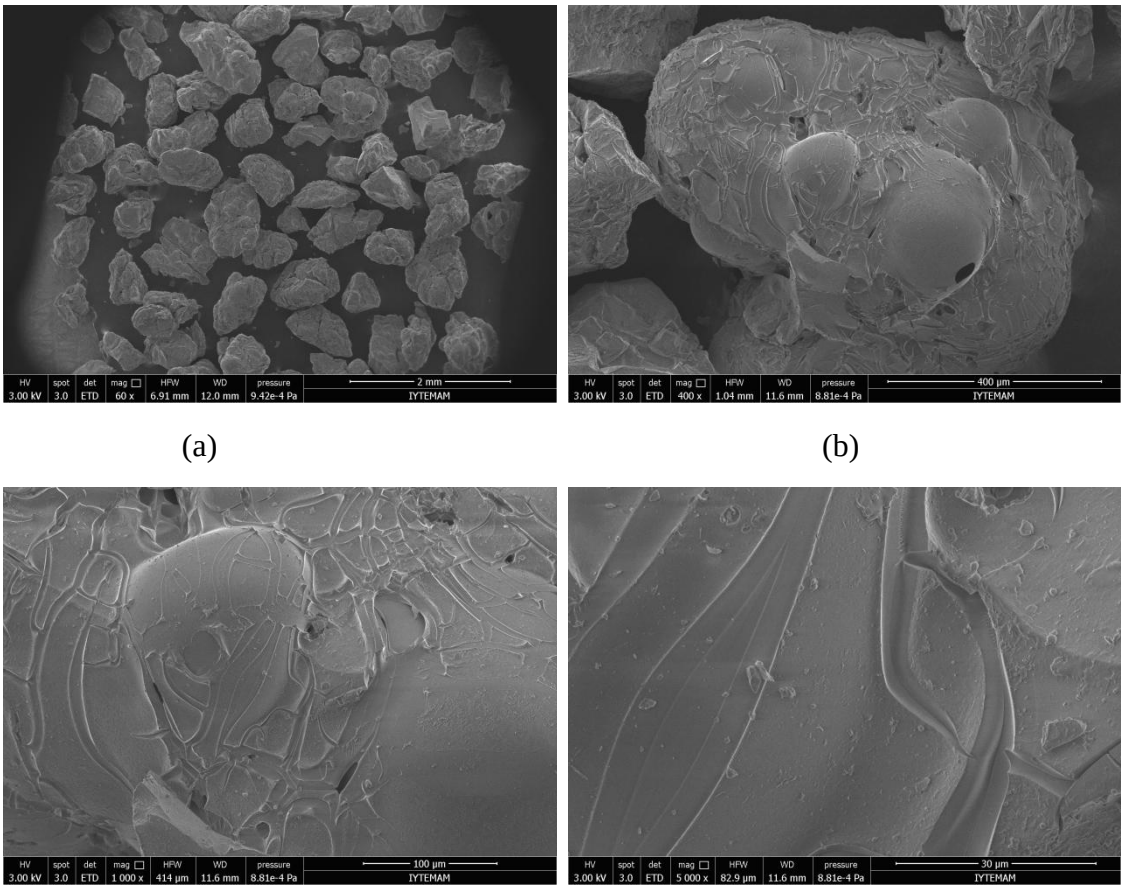
Figure 3.13.d. Raw perlite SEM images from Mollatepe samples taken at different magnifications: (a) 100x, (b) 500x, (c) 1000x, (d) 2500x.



(a) (b)

(c) (d)

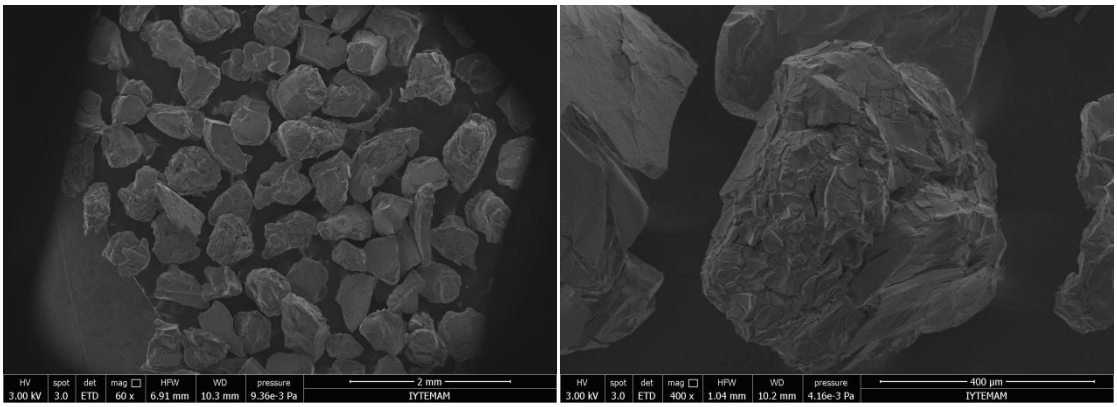
Figure 3.14. Expanded perlite SEM images from Bergama 01 samples taken at 1000 ˚C magnifications: (a) 60x, (b) 400x, (c) 1000x, (d) 5000x.



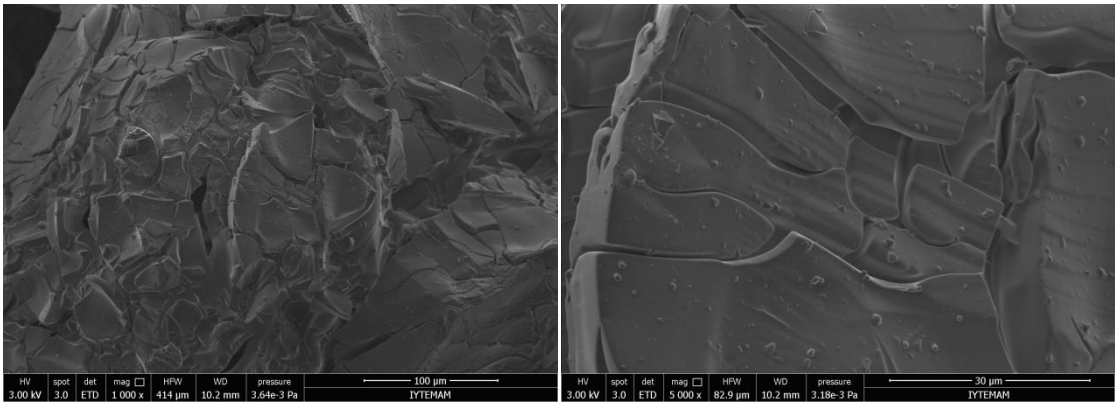
(a) (b)

(c) (d)

Figure 3.15. Expanded perlite SEM images from Bergama 02 samples taken at 1000 ˚C magnifications: (a) 60x, (b) 400x, (c) 1000x, (d) 5000x.

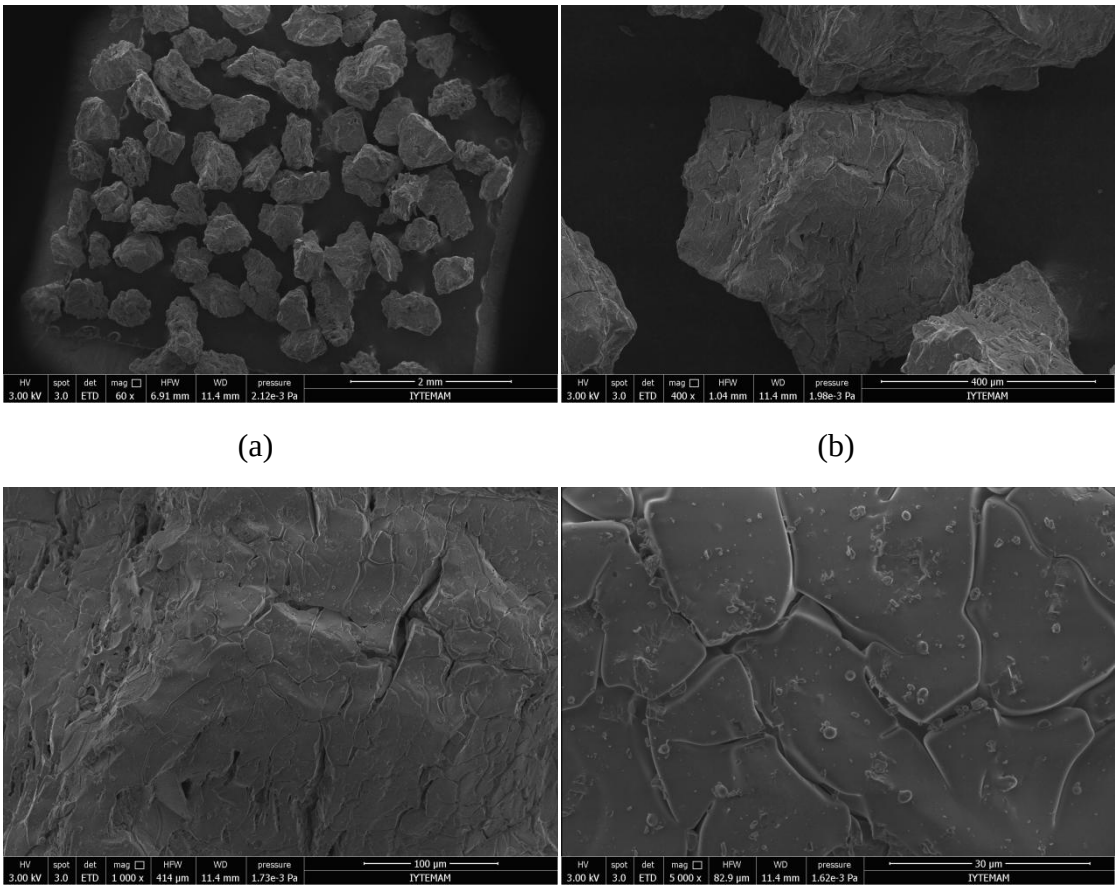


(a) (b)



(c) (d)

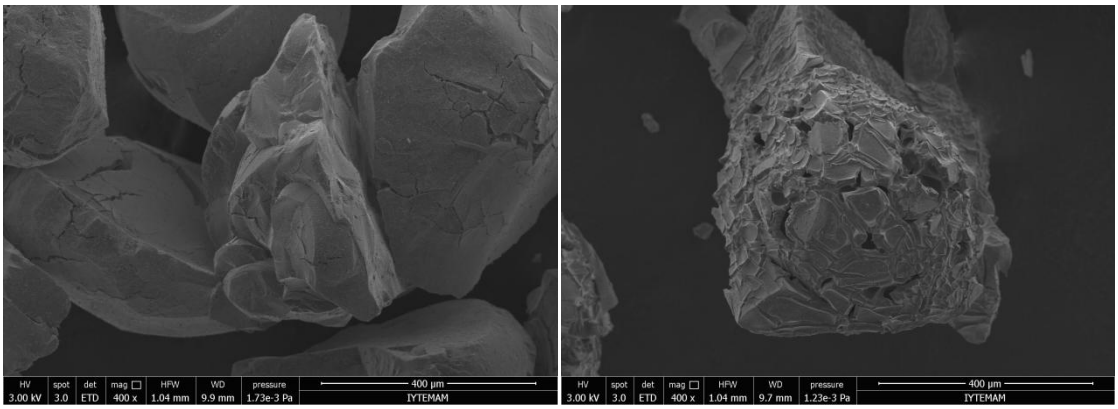
Figure 3.16. Expanded perlite SEM images from Avdan samples taken at 1000 ˚C magnifications: (a) 60x, (b) 400x, (c) 1000x, (d) 5000x.



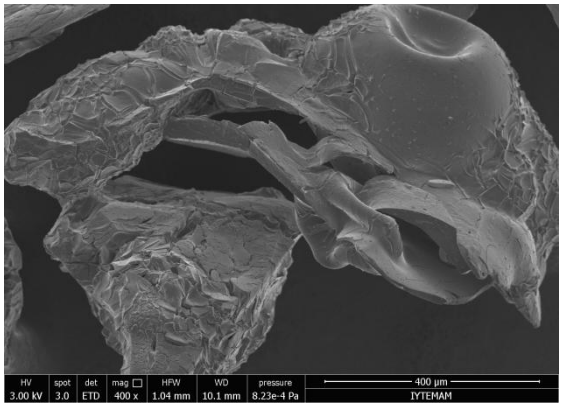
(a) (b)

(c) (d)

Figure 3.17. Expanded perlite SEM images from Mollatepe samples taken at 1000 ˚C magnifications: (a) 60x, (b) 400x, (c) 1000x, (d) 5000x.

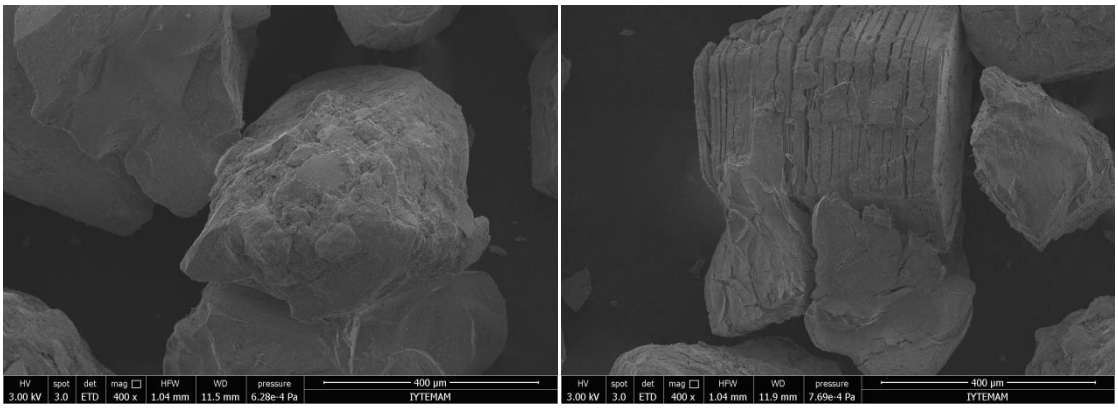


(a) (b)

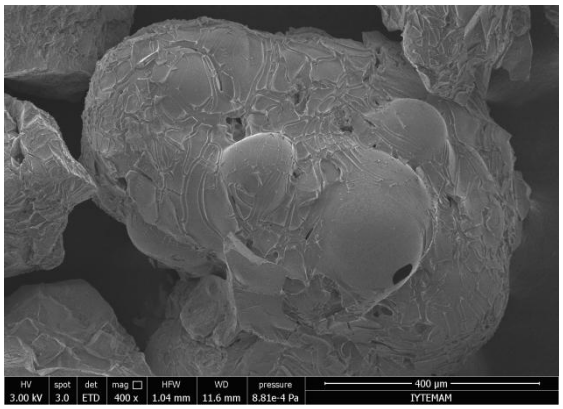


(c)

Figure 3.18. Expanded perlite SEM images from Bergama 01 samples taken at 400x magnifications: (a) 600 ˚C, (b) 800˚C, (c) 1000˚C.

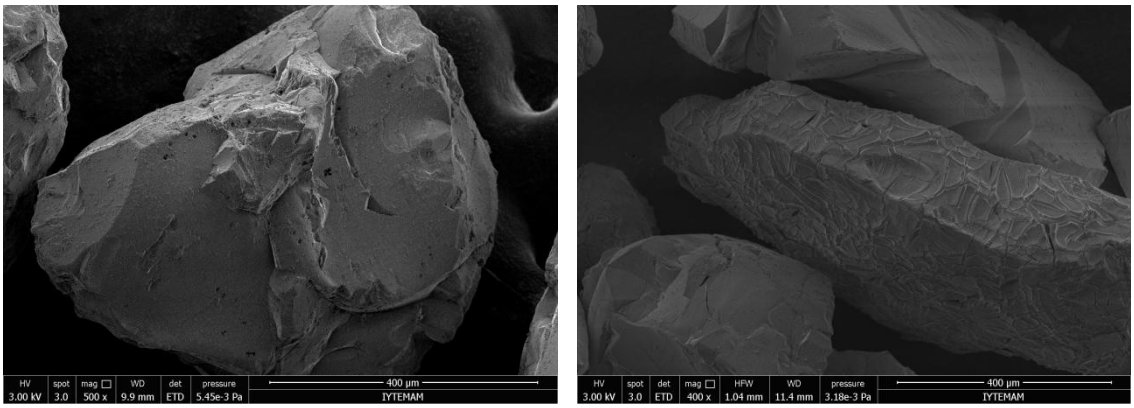


(a) (b)

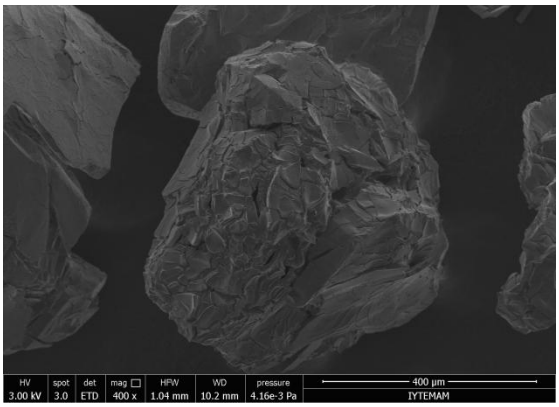


(c)

Figure 3.19. Expanded perlite SEM images from Bergama 02 samples taken at 400x magnifications: (a) 600 ˚C, (b) 800˚C, (c) 1000˚C.

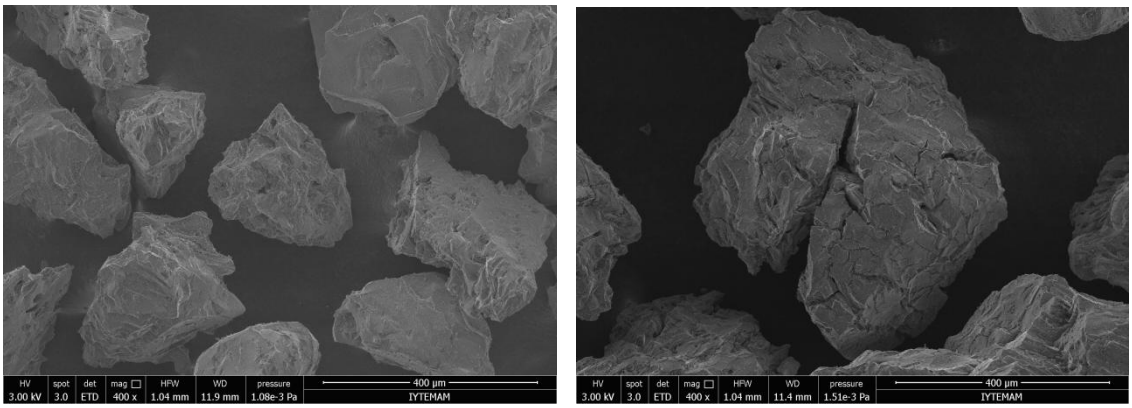


(a) (b)

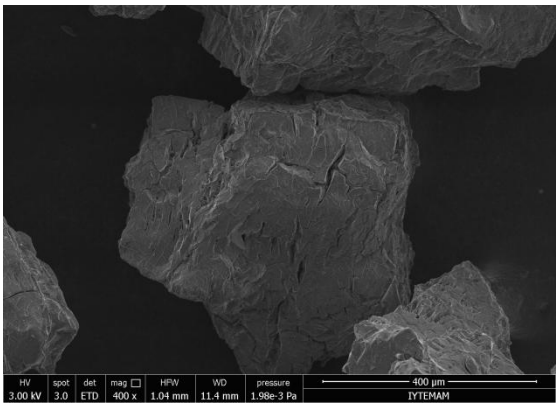


(c)

Figure 3.20. Expanded perlite SEM images from Avdan samples taken at 500x-400x-400x magnifications: (a) 600 ˚C, (b) 800˚C, (c) 1000˚C respectively.



(a) (b)



(c)

Figure 3.21. Expanded perlite SEM images from Mollatepe samples taken at 400x magnifications: (a) 600 ˚C, (b) 800˚C, (c) 1000˚C.

As far as firstly no cracks on the surface and the grain is smooth superficially but after the heat treatment many cracks formed on the surface so that indicates the expanded (Figures 3.15.-18.) It was given SEM images of expanded perlite samples (Figures 3.15. to 3.18.). These the SEM images of expanded perlite is at 1000˚C due to much more expansion at 1000˚C for all the samples (Table 3.6.), magnifications at 60x, 400x, 1000x, 5000x and particle size is 400-500 µm. Also, although the raw perlite samples have similar properties, the expansion behaviors are so different because of forming the perlite e.g. the temperature and viscosity of lava. If the lava cools slowly happening more crystal formation (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006).

After heat treatment (at 1000˚C), it was occurred so many cracks and porous (Figures 3.14 to 17). Figure 3.14.(a) was observed that most particles expanded and occured more cracks, open and close porous and well dispersed voids (b), (c), (d) than others.

Figure 3.15. showed that most particles were expanded (a), slightly blows up the grain as shown by the cracks developed on the surface (b), besides the blows up small close porous formation on the smooth surface (c),(d). Figure 3.16. showed that most particles were expanded too (a), but not open porous and blows up, formation close porous (b), (c), (d). Figure 3.17.showed that some particles were expanded (a), formation cracks somewhere (b), (c), formation close porous (d).

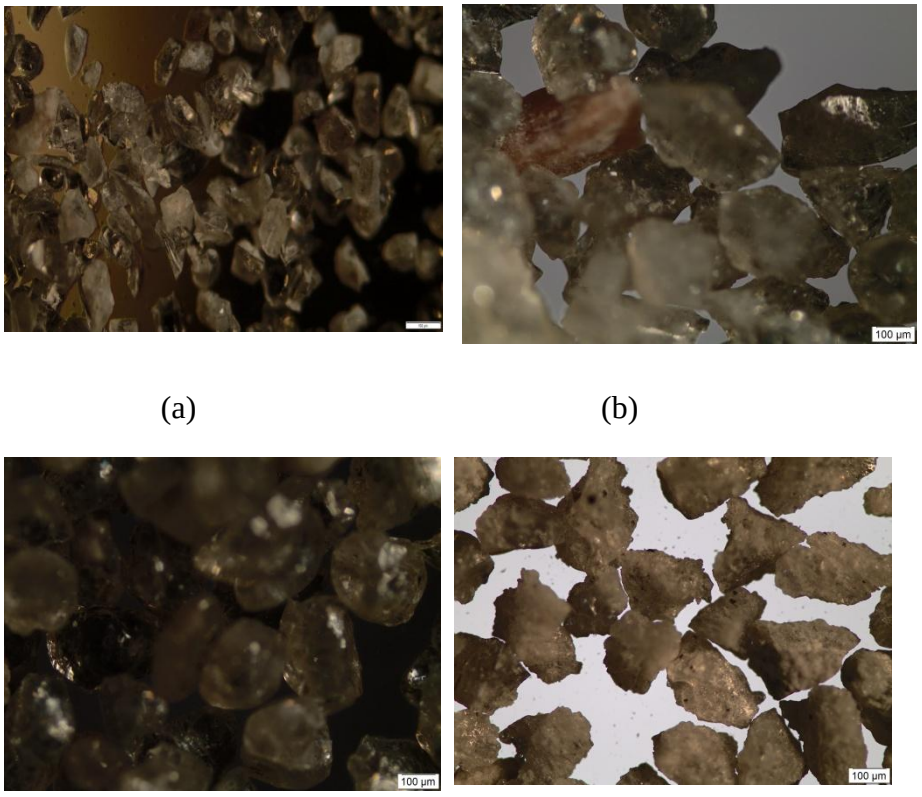
Figures 3.18.to 3.21. showed that SEM micrographs of expanded perlite samples from Bergama 01, Bergama 02, Avdan and Mollatepe, respectively at 600˚C, 800˚C, 1000˚C, 400x magnifications. As seen that SEM images of the expanded perlite samples (in Figures 3.18. to 21.) it was observed much more porous formation increasing temperatures. Figure 3.18. showed that almost no less expansion (a), formation so many cracks and small open porous (b), more than open and close porous and formation blows up the grain (c). Figure 3.19. showed that most of the particles smooth (a), started many cracks on the surface (b), formation close porous and blows up the grain(c). Figure 3.20. showed that almost all of the particles smooth on the surface due to no expansion (a), some particles formation many cracks and close porous(b), more cracks and close porous(c). Figure 3.21. showed that no expansion like raw perlite samples (a), inner layers tries to escape with many cracks (b), formation many cracks and close porous (c).

As a result, according to the SEM images of the perlite samples we can see that changed grain morphologies of perlite samples. While perlite is a volcanic rock, it has become porous by expanding.

It also has been taken images from raw and expanded perlite samples in each mine with optical microscopy. These images of the raw perlite samples were formed as incident and transmitted, magnifications 4x and 10x in Figures 3.22.-3.23., respectively. After volcanic eruption, perlite samples released uncontrolled, it may contain different elements that are not in of itself. It can be seen the dark points in figures that it proves the existence of these elements (Figures 3.22 to 23.).

It was showed that the images of expanded perlite samples (Bergama 01) as incident and transmitted, (magnifications 10x) in Figures 3.24. to 25., respectively. It can be seen that expansion increased with temperature. Figure 3.24. showed the expansion of particles were increasing (a), (b), (c), many bubbles also small holes (d). As seen that the Figure 3.25. increasingly closed porous were observed (a), (b), (c), the biggest porous was observed at 1200 ˚C (d). It was showed that the images of expanded perlite samples (Bergama 02) as transmitted, magnifications 10x in Figures 3.26. less amount cracks (a), formation cracks and closed porous (b), more bigger closed porous (c). It was showed that the images of expanded perlite samples (Avdan) as transmitted, magnifications 10x, in Figure 3.27. very small cracks due to less expansion (table 3.5.) (a), formation of small cracks (b), bigger cracks and closed porous (c). It was showed that the images of expanded perlite samples (Mollatepe) as transmitted, magnifications 10x in Figure 3.28. not formation cracks due to no expansion (a), uncontrolled cracks dispersed and started some closed porous (b) and (c).

Also it was shown which are the images of expanded perlite samples from Mollatepe, Avdan and Bergama (Bergama 02) regions in Figures 3.24. to 28. could not be obtained at 1200 ˚C due to the adhesion of the crucible. It was observed that much more porous and cracks occurs increasing temperature.



(a) (b)

(c) (d)

Figure 3.22. Incident Light Optical Microscopy images of raw perlite sample regions from Bergama (Bergama 01), (Bergama 02), Avdan, Mollatepe magnifications 4x are shown in (a), (b), (c), and (d), respectively.

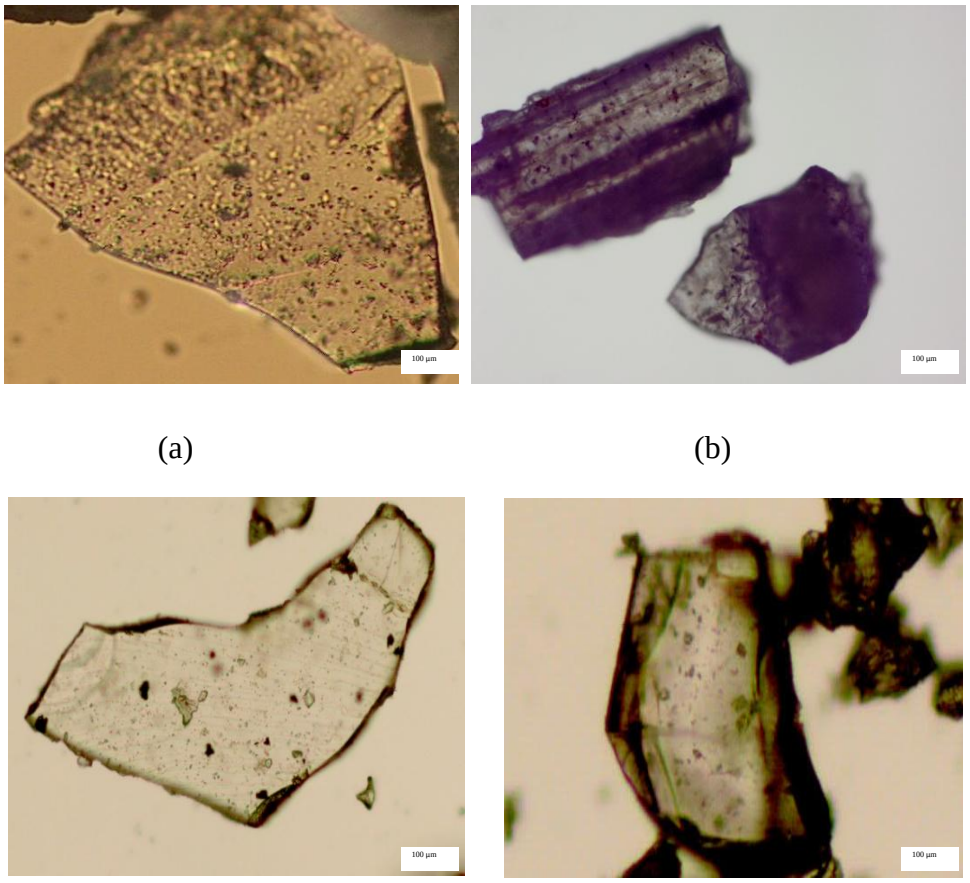
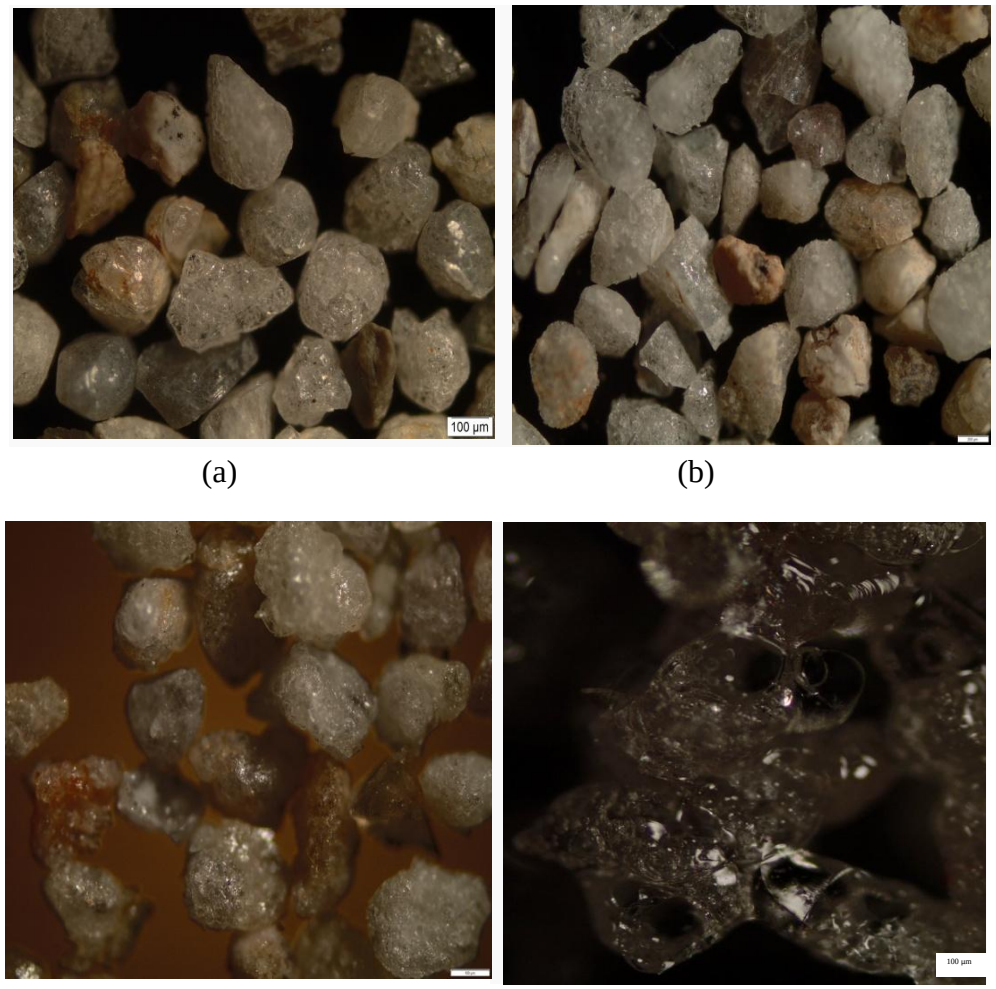


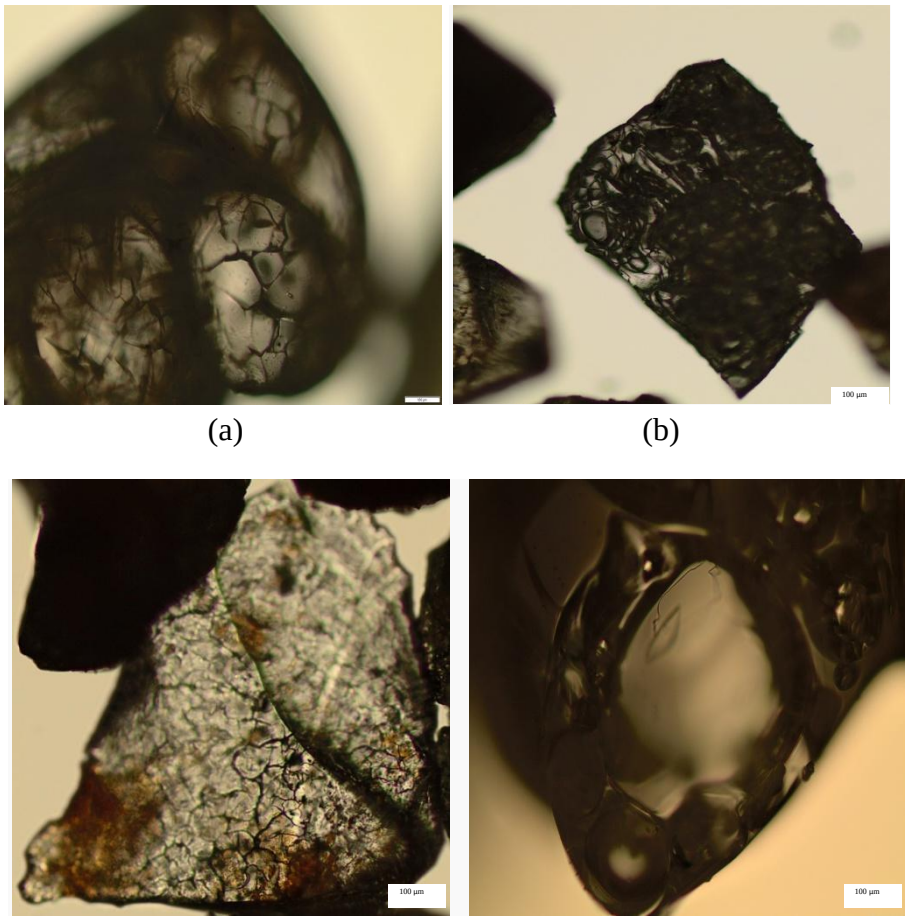
Figure 3.23. Transmitted Light Optical Microscopy images of raw perlite sample regions from Bergama (Bergama 01), (Bergama 02), Avdan, Mollatepe magnifications 10x are shown in (a), (b), (c), and (d) respectively.



(a) (b)

(c) (d)

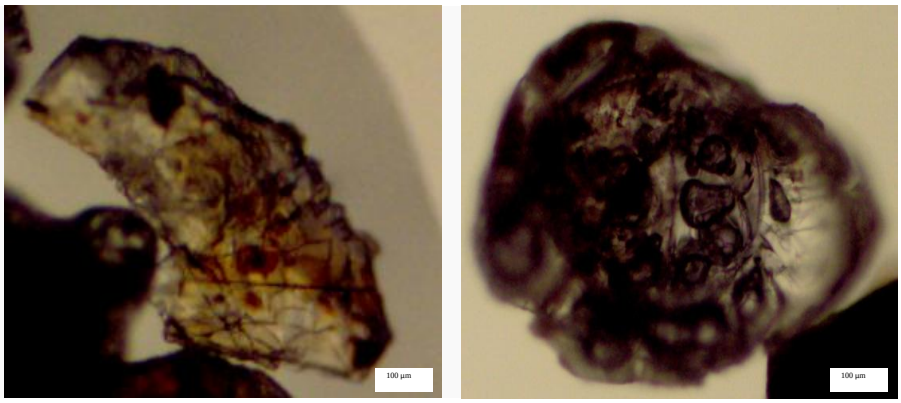
Figure 3.24. Incident Light Optical Microscopy images of Bergama 01 expanded perlite samples magnifications 10x, at (a) 600˚C, (b) 800˚C, (c) 1000˚C and (d) 1200˚C.



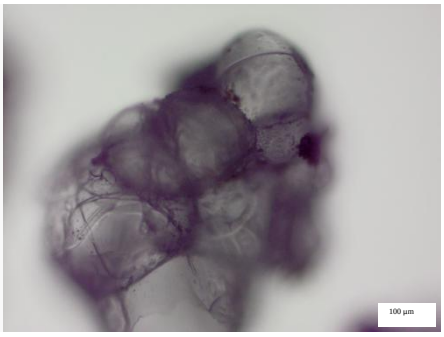
(a) (b)

(c) (d)

Figure 3.25. Transmitted Light Optical Microscopy images of Bergama 01 expanded perlite samples magnifications 10x, at (a) 600˚C, (b) 800˚C, (c) 1000˚C and (d) 1200˚C.

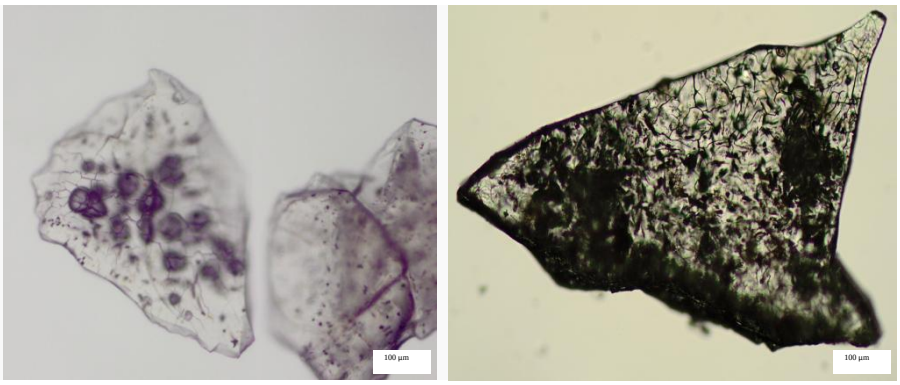


(a) (b)

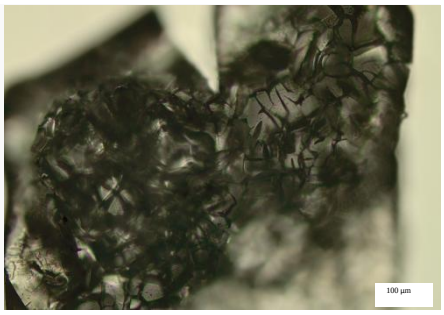


(c)

Figure 3.26. Transmitted Light Optical Microscopy images of Bergama 02 expanded perlite samples magnifications 10x, at (a) 600˚C, (b) 800˚C, and (c) (in water) 1000˚C.

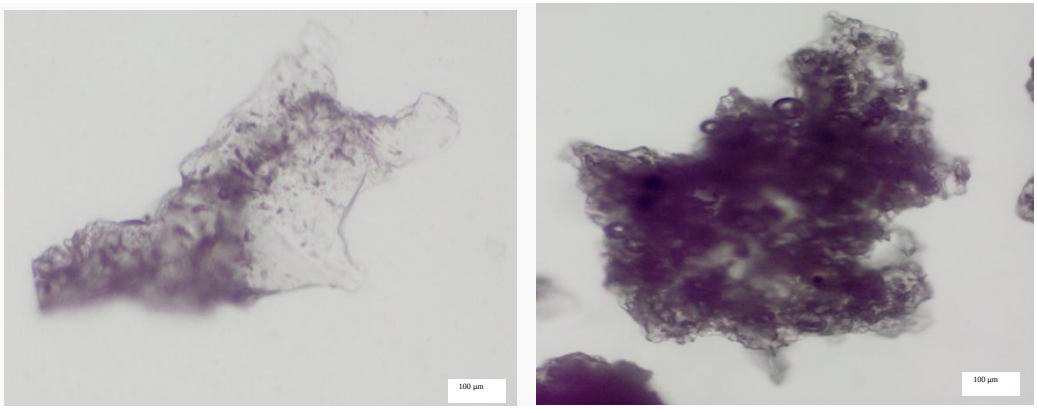


(a) (b)

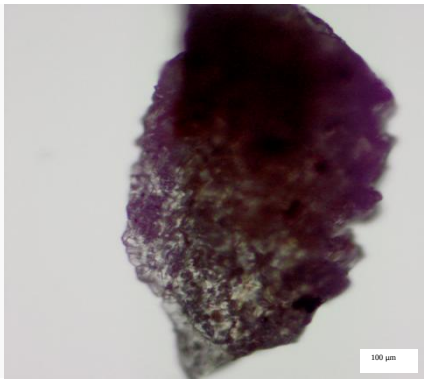


(c)

Figure 3.27. Transmitted Light Optical Microscopy images of Avdan expanded perlite samples magnifications 10x, at (a) (in water) 600˚C, (b) 800˚C, and (c) 1000˚C.

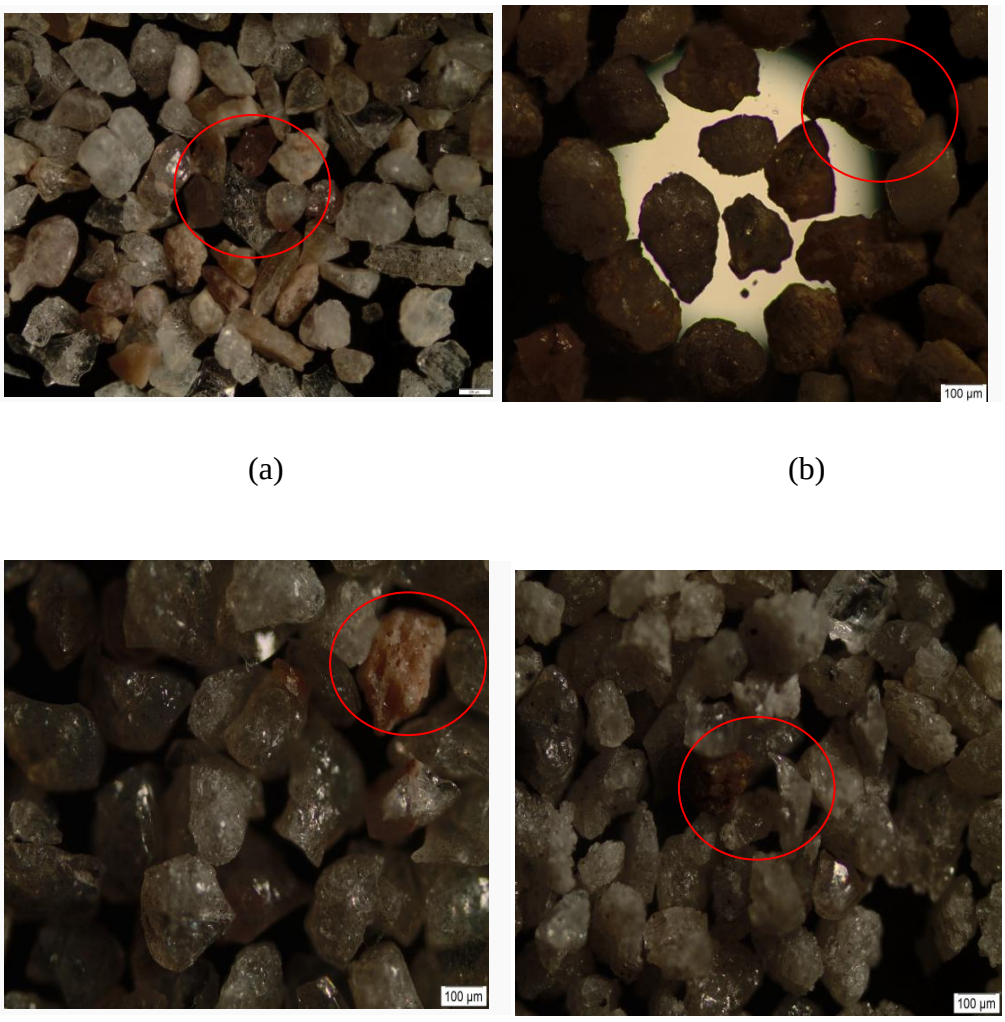


(a) (b)



(c)

Figure 3.28. Transmitted Light Optical Microscopy images of Mollatepe expanded perlite samples magnifications 10x, in water at (a) 600˚C, (b) 800˚C, and (c) 1000˚C.



(a) (b)

(c) (d)

Figure 3.29. Incident Light Optical Microscopy images of (a) Bergama 01, (b) Bergama 02, (c) Avdan and (d) Mollatepe expanded perlite samples magnifications 4x at 800˚C.

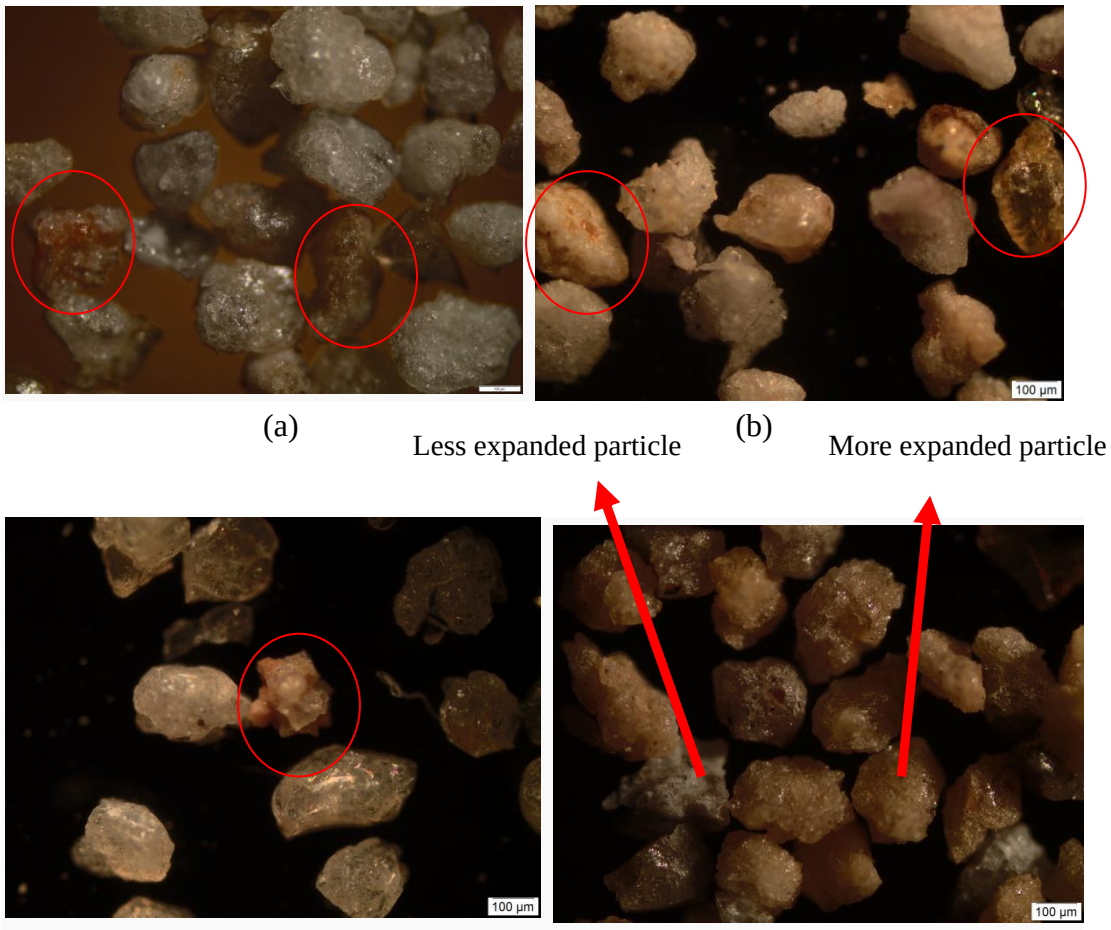


Figure 3.30. Incident Light Optical Microscopy images of (a) Bergama 01, (b) Bergama 02, (c) Avdan and (d) Mollatepe expanded perlite samples magnifications 10x at 1000˚C.

Figures 3.29. to 30. showed the optical images of the expanded perlite samples as incident, magnifications 4x and 10x at 800 ˚C and 1000 ˚C , respectively.

In generally, the expansion effect caused to closed of porous formation. Moreover, some samples were expectedly expanded, while some of them were not expanded or partially expanded. This outcome causes both uncontrolled released of perlite samples in volcanic eruptions and consisting of different chemical compositions. Figure 3.30. can be given as an example to understand this variation. Additionally, the amount of iron composition in perlite cause the color change in sample. The more iron content leads to decreasing of whiteness as linearly (Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite, 1999), (SAKKA, Sodeyama, & Furubayashı, 2000). Lastly, the colors of raw perlite and expanded perlite are grey and white with

related to the iron content in the perlite sample (shown by round take into, see fig. 3.29. to 30.).

Although SEM has higher resolution imaging to analyzing of morphological structure, even not give certain results was reached similar images by the optical microscopy. Figure 3.31. to 38. show that the similar images of SEM and optical microscopy for all of the raw and expanded perlite samples. It was showed the images all of the expanded perlite samples at 1000˚C.

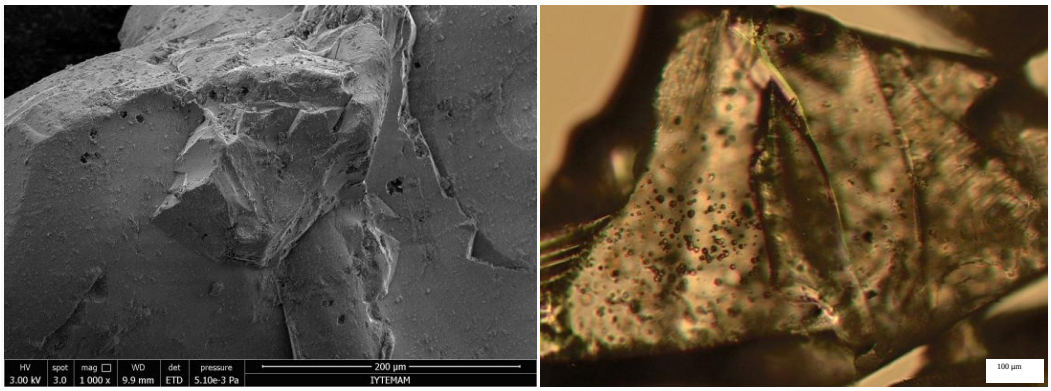


Figure 3.31. SEM and Transmitted Light Optical Microscopy images of Bergama 01 raw perlite samples magnifications 1000x and 10x, respectively.

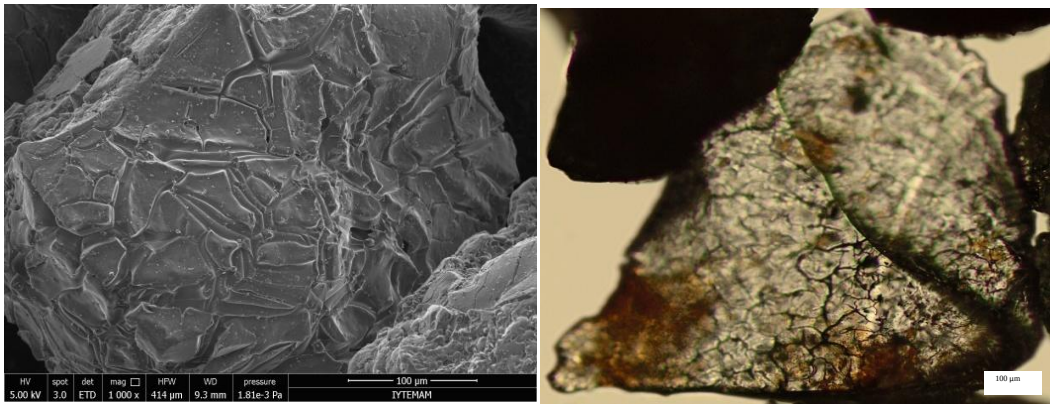


Figure 3.32. SEM and Transmitted Light Optical Microscopy images of Bergama 01 expanded perlite samples at 1000 ˚C, magnifications 1000x and 10x, respectively.

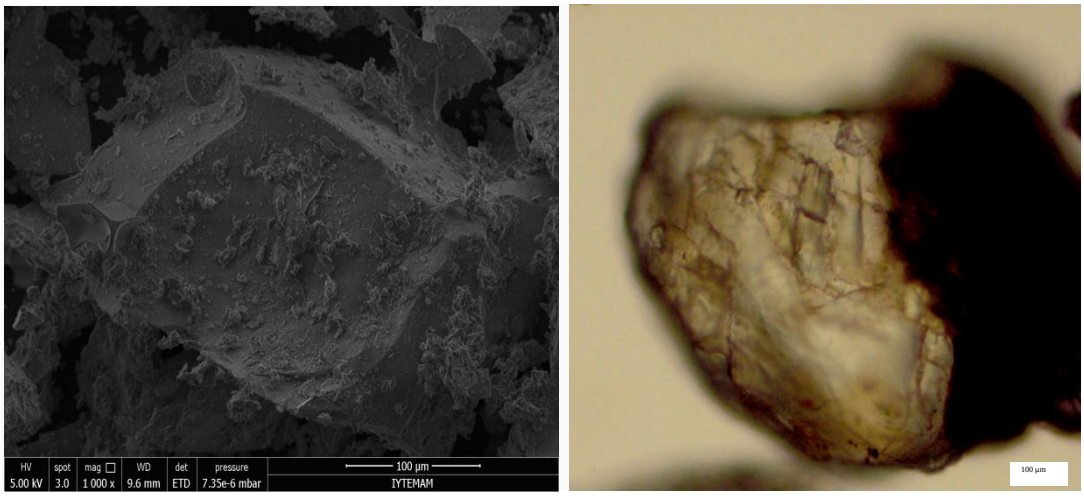


Figure 3.33.SEM and Transmitted Light Optical Microscopy images of Bergama 02 raw perlite samples magnifications 1000x and 10x, respectively.

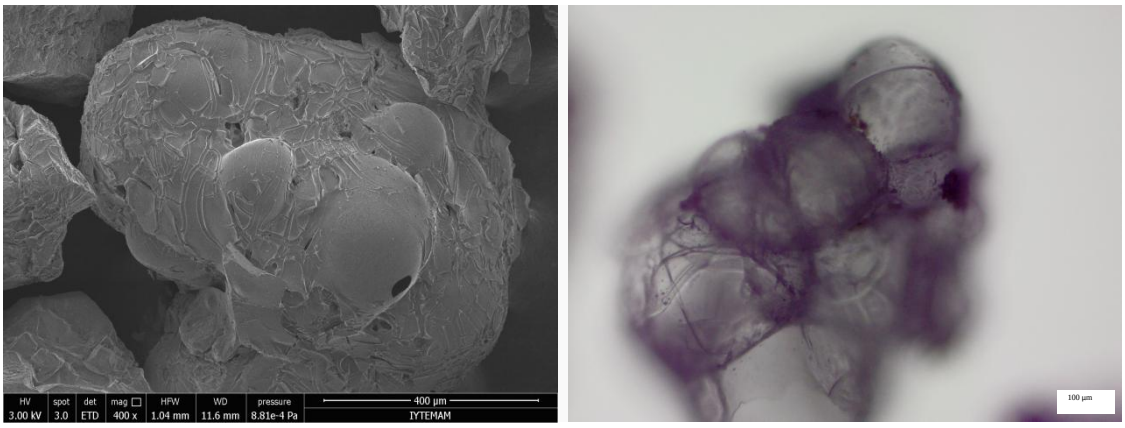


Figure 3. 34. SEM and Transmitted Light Optical Microscopy images of Bergama 02 expanded perlite samples at 1000 ˚C, magnifications 400x and 10x, respectively.

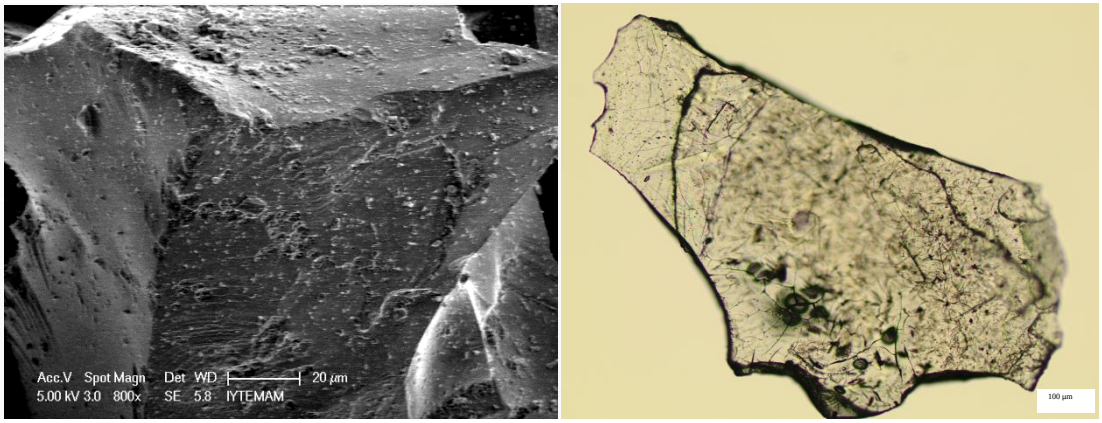


Figure 3.35. SEM and Transmitted Light Optical Microscopy images of Avdan raw perlite samples magnifications 800x and 10x, respectively.

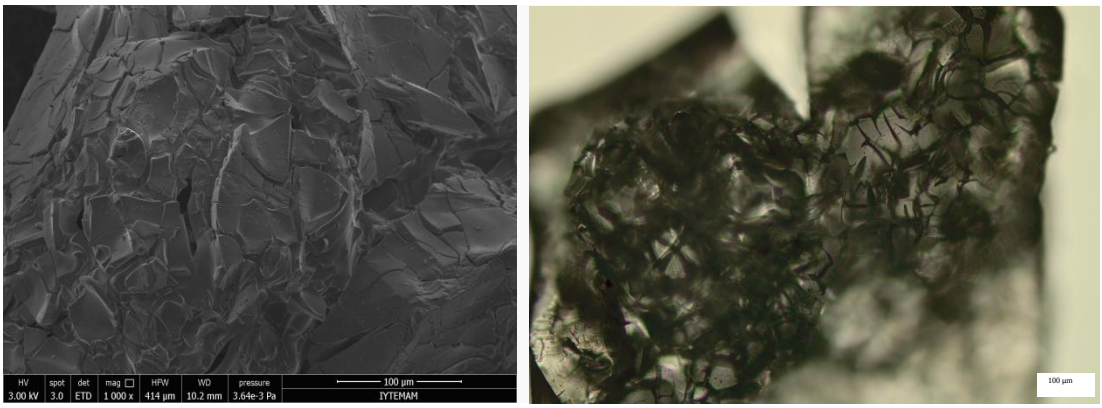


Figure 3.36. SEM and Transmitted Light Optical Microscopy images of Avdan expanded perlite samples at 1000 ˚C, magnifications 1000x and 20x, respectively.

100 µm

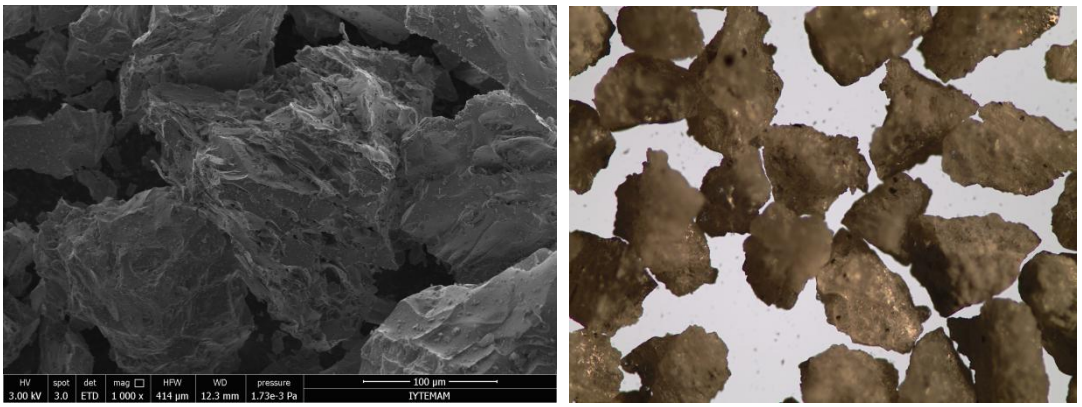


Figure 3.37. SEM and Incident Light Optical Microscopy images of Mollatepe raw perlite samples magnifications 1000x and 10x, respectively.

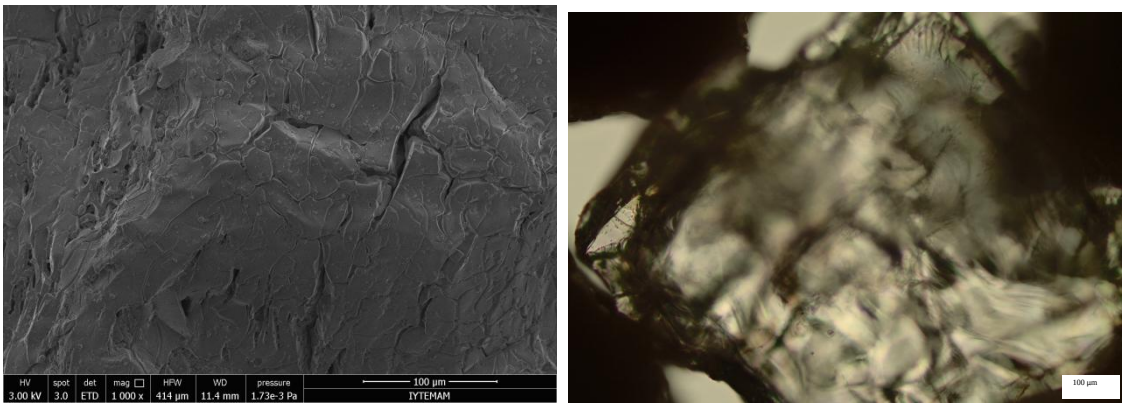


Figure 3.38. SEM and Transmitted Light Optical Microscopy images of Mollatepe expanded perlite samples at 1000 ˚C, magnifications 1000x and 20x, respectively.

3.3. Discussion of Expansion Behaviour of Perlite

Perlite in its expanded form has a higher economic value due to its porous structure giving the material its light weight and thermal insulation properties. The expansion of perlite is a common in commercial practice where the process simply takes place in vertical ovens providing a heating profile to the perlite grains (Angelopoulos, Gerogiorgis, & Paspaliaris, 2013). This heating allows perlite to attain its porous structure. The purpose of this study is to characterize the properties of raw perlite and use the features defined in this characterization as it pertains to their effects on the expansion of perlite to help provide a definition on how and when perlite expands. The study has used raw perlite from four different sources in order to determine any differences or correlations that may be present on the ability of these materials to expand.

Generally, the factors affecting expansion are temperature, grain size, time, and water released from the material. In addition, the presence and amount of crystalline phase in perlite is also known to effect perlite expansion. Water release plays an important role as other factors affecting expansion (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006) (Jing, Fang, Liu, & Liu, 2011). Sodeyama and Sakka (Sodeyama & Sakka, 2005) suggest that the suitable amount of water for expansion is between 1.5-2.0 %. Jing et al. expansion (Jing, Fang, Liu, & Liu, 2011) further suggest that if water content in raw perlite is excessive cracks can occur due to formation of high vapor pressure during the expansion process. They, therefore, propose a preheating treatment to remove the excess water. The perlite samples taken from these different regions expansion behavior varies according to water release. Similarly different cracks occurred in each grains showed that expansion rates of the perlite samples were varied in each different mine.

Using XRD analysis, perlite from the four sources was determined to predominantly possess an amorphous structure. The amount of the glassy phase was determined to be at 10 % and below, depending on the source of the raw material. the findings in this study, in parallel to the results obtained from other studies (Sodeyama, Sakka, & Kamino, Preparation of Fine Expanded Perlite, 1999), (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006), show that the presence of crystal phase have a significant effect on defining the ability of the raw perlite to expand. Accordingly,

samples from Bergama 02 and Mollatepe, which show presence of crystalline phase, did not expand as much as the Bergama 01 and Avdan samples which showed the least amount of crystal phase.

The chemical composition results determined using XRF analysis showed the overall material to be predominantly formed of SiO_2, Al_2O_3, K_2O, Na_2O, Fe_2O_3, and CaO. The glass phase of the raw material was, therefore, deemed to possess an alkali-alkali earth-alumino-silicate based chemistry.

The TGA results showed the weight loss due to heating of the raw samples to range between 1.5 to 2.5% within a temperature range of 190-650 ˚C for all four samples. Both the total amount of weight reduction and its temperature range relates to the release of physisorbed and chemisorbed water and their ultimate influence on the ability of raw perlite to expand. The expansion of perlite was shown to create a porous structure formed of closed and open cell pores. This was true for grains that showed ability to expand. This, therefore, suggests that not all particles that would possess water will have a tendency to expand. The ability and range of temperatures to release the water from the structure is an important property of the material. Roulia (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006) has reported that upon heat treatment within 0-250 ˚C 20-45 % of the total water content in perlite is released, which she has attributed this to the removal of molecular water. We here refer to this water as physisorbed water. At 250-550 ˚C temperature range, Roulia suggests that the material starts to soften, releasing 45-65 % of its total water content, which she attributes to the release of -OH groups. She also suggests that some molecular (physisorbed) water which may have been trapped in the inner closed pores of the material starts to evaporate and pushes its way out within this higher temperature range. At 550-950 ˚C temperature range, Roulia shows that the perlite releases the remaining 5-20 % of its total water content.

In addition to the ability of water release discussed above, the findings in this study, in parallel to the results obtained from other studies, (Roulia, Chassapis, Kapoutsis, Kamitsos, & Savvidis, 2006) show that the presence of crystal phase have a significant effect on defining the ability of the raw perlite to expand. Accordingly, samples from Bergama 02 and Mollatepe, which show presence of crystalline phase, did not expand as much as the Bergama 01 and Avdan samples which showed the least amount of crystal phase.

The other important factor described from the results found in this study is the temperature at which the expansion starts and concludes. Laboratory expansion tests suggest a difference between the four samples tested in this study. These findings are found to correlate well with the presence and amount of crystalline phase in the raw material where Bergama 02 sample with the highest level of crystal phase showed a lowest expansion capability followed by Mollatepe sample, which had lower amount of crystal phase relative to Bergama 02. In addition, the temperature where the raw perlite is heat treated is also seen to play an important role on the degree of expansion. The effects of temperature can be attributed to the glass composition and, therefore, its structure, which in turn affects the viscosity of that glass. The ability of the glass to flow will allow for the nucleation and growth of the bubbles and the creation of the porous structure of the expanded perlite.

In addition to the expansion tests carried out in the laboratory, expanded perlite samples obtained from commercial practice was also characterized for its morphology using SEM. Figure 3.39. shows the images of commercially expanded perlite. A comparison of the commercially expanded particle with perlite expanded in the laboratory shows the differences between the two materials (Figures 3.39. with 3.14-15-16-17-18-19-20-21.). The commercial expanded perlite shows an open network of pores. These pores are linked to each other through a very thin skin, which appears to be very fragile. In comparison, the laboratory expanded perlite shows a closed morphology on the surface, with the optical microscopy images taken from similar grains showing the internal structure of such particle to possess a closed cellular network. The key difference between these two processes can be attributed to the heat treatment duration where the laboratory process has subjected the material to a total of 5 minutes of heat whereas the commercial samples see the heat application within few seconds (Gürtürk, Oztop, & Hepbaslı, Comparison of Exergoeconomic Analysis of Two Different Perlite, 2015), (Klipfel, Founti, Zahringer, Martin, & Petit, 1998). The difference in the morphology based on the heat treatment durations suggest that the laboratory process has allowed for the grains to melt and close any open pores while the short heat application has given room for pores to expand and explode. This implies that a slower heating rate will allow for less expansion, which has also been reported by Aguilar et al. (Aguilar-Garib, García-Onofre, Ortiz, & Valdez-Nava, 2013).

In addition to aluminosilicate glasses have a property of high glass transition temperature and softening point. Perlite samples softening points varies between 871-1093 ˚C (Perlite Institute, 2011) and according to (Souza & Martinelli, 2015) the transition temperature of aluminosilicate glass has a temperature variation of 808-818 ˚C. Expansion of perlite starts above the glass transition temperature with the perlite particles showing increased tendency to sinter at temperature range between 1000-1090 ˚C (Aguilar-Garib, García-Onofre, Ortiz, & Valdez-Nava, 2013), (Souza & Martinelli, 2015). It is known that glasses are fluid above their glass transition temperature with its fluidity increasing with increasing temperature (Varshneya, 1994). The increased fluidity (also referred to as viscosity) of the material allows for increased diffusion which causes the particles that contact each other under such conditions to sinter and adhere. Accordingly, the softening temperature of perlite can be considered as a reference point where increased sintering kinetics is present and particles are prone to adhesion. The particles in the laboratory experiments shown and discussed in this study have all been kept at a stagnant condition allowing an opportune condition for the particles to sinter and adhere during the heat treatment experiments. This will influence the pore formation and growth by allowing the particles as well as the formed pores to coalesce. This effect is expected to be significant above the softening temperature. In accordance with these factors, experiments conducted at 1020 ˚C, 1050 ˚C, 1065 ˚C and 1200 ˚C temperatures, which are above the softening temperature, have all demonstrated the adhesion of the perlite particles (see Figures 3.40. - 41.). Expansion results and analysis was, therefore, not possible at temperatures above 1000 ˚C. In order to avoid particle adhesion at these temperatures, the particles should be suspended without touching each other. This is what the particles experience in the industrial production (Sodeyama & Sakka, 2005). Also, as a model in the form of a summary the stages of expansion of perlite are given in section 4. For future study, the ability for expansion above the softening temperature can be demonstrated by exposing the particles to a condition where they are not in contact with each other.

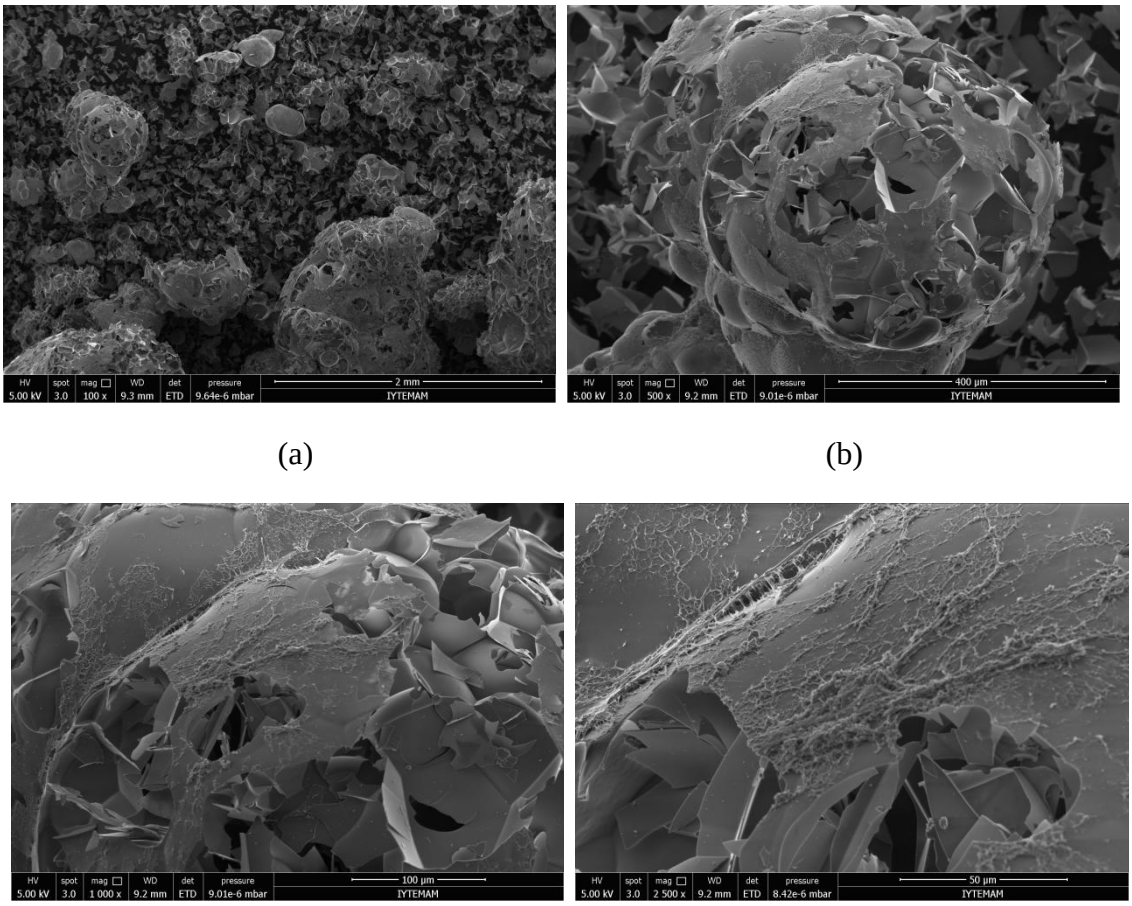
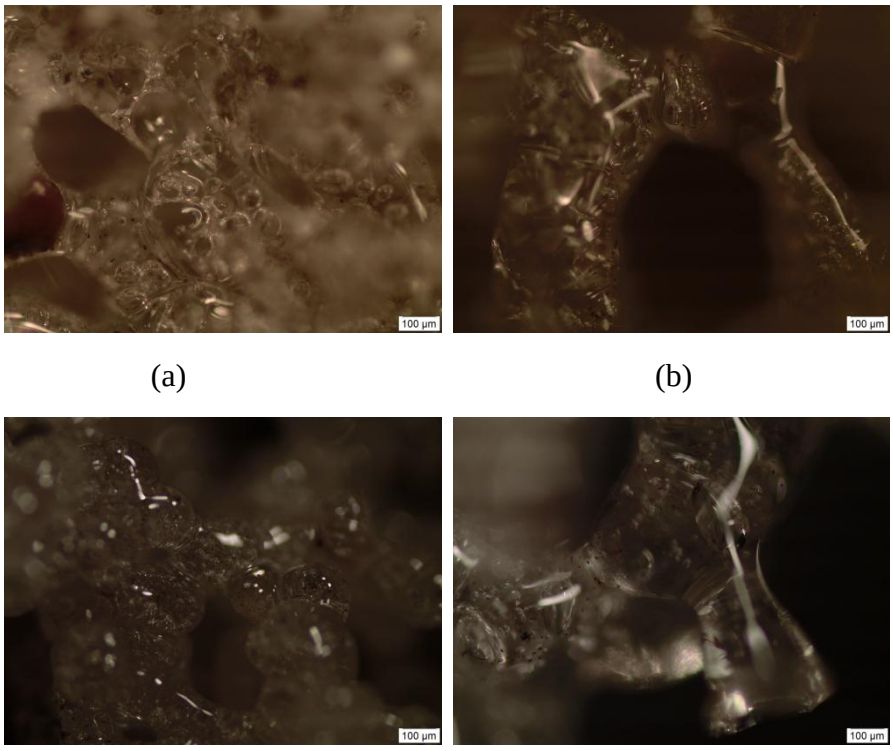


Figure 3.39. SEM images of perlite samples (Bergama 03) were expanded by Çullas Group in Bergama Mine (magnifications 100x, 500x, 1000x and 2500x, respectively).

(a) (b)

(c) (d)

Figure 3.40. Expanded perlite samples at (a) 1020 ˚C, (b) 1050 ˚C, (c) 1065 ˚C and (d) 1200 ˚C, respectively.



(a) (b)

(c) (d)

Figure 3.41. Incident Light Optical Microscopy images of perlite samples at (a) 1020˚C, (b) 1050˚C, (c) 1065˚C, (d) 1200˚C (magnifications 10x).

CHAPTER 4

CONCLUSIONS

In this master thesis, four different perlite samples were characterized for their critical properties and the expansion behaviours of perlite samples were analyzed. Samples which were obtained from Bergama, Avdan, and Mollatepe regions of Turkey were characterized and their expansion behaviour of was investigated The characterization of raw perlite and expanded perlite samples were determined by XRD, XRF, TGA, SEM and optic microscope. Four different heat treatments at 600˚C, 800˚C, 900˚C and 1000 ˚C and four different sizes 400-500 μm, 315-400 μm, 200-315 μm and 160-200 μm were characterized.

Chemical analysis of perlite samples determined using XRF analysis showed to formed of SiO_2, Al_2O_3, K_2O, Na_2O, Fe_2O_3, and CaO and consistent with typical chemical composition of perlite. Also it showed that this glass is of an alkali-alkali earth-alumino-silicate based chemistry.

The XRD analysis showed crystalline and amorphous structure of perlite samples. The results of XRD patterns showed that found crystal amount between 1%-11% with the highest value measured from Bergama-02. According to the information obtained from the literature the amount of crystal in the perlite affects the expansion process and the expansion analysis results in this study confirm that the amount of the crystal affected the expansion of more or less.

Another factor affecting the expansion of perlite water release. The chemisorbed water release occurs above the glass transition temperature range and the physisorbed water is as molecular water in perlite. The results of TGA analysis revealed that the water release above the glass transition temperature of perlite to occur and it was concluded that the results are found to be consistent with the literature.

The results of laboratory experiments observed the most expansion ratio Bergama 01 (approximately 2 times exp.), following Avdan (approximately 1.5 times exp.) according to changes the volume and density of perlite samples. These results are consistent with XRD results (presence amount of crystal in the perlite).

The morphologies all of the perlite samples were determined using SEM and optical microscopy. All of the raw perlite samples showed smooth fracture surface, containing cracks and inclusions. After heat treatment, perlite samples showed depending on temperature the formation of a combination of cracks, closed, open pores and flow. SEM images show the formations of up to 1000 ˚C. SEM images of the perlite samples could not show in this study above the softening temperatures due to forming sinter and adhesion, but the expansion carried out at temperature above 1000 ˚C in industrial furnaces. The expanded perlite samples in industrial furnace have more porous structure than the expanded perlite samples in the laboratory. Figure 3.42. showed that the stages of perlite samples expansion briefly. If the expansion conditions are carried out controller manner, it can be reached the hollow particles.

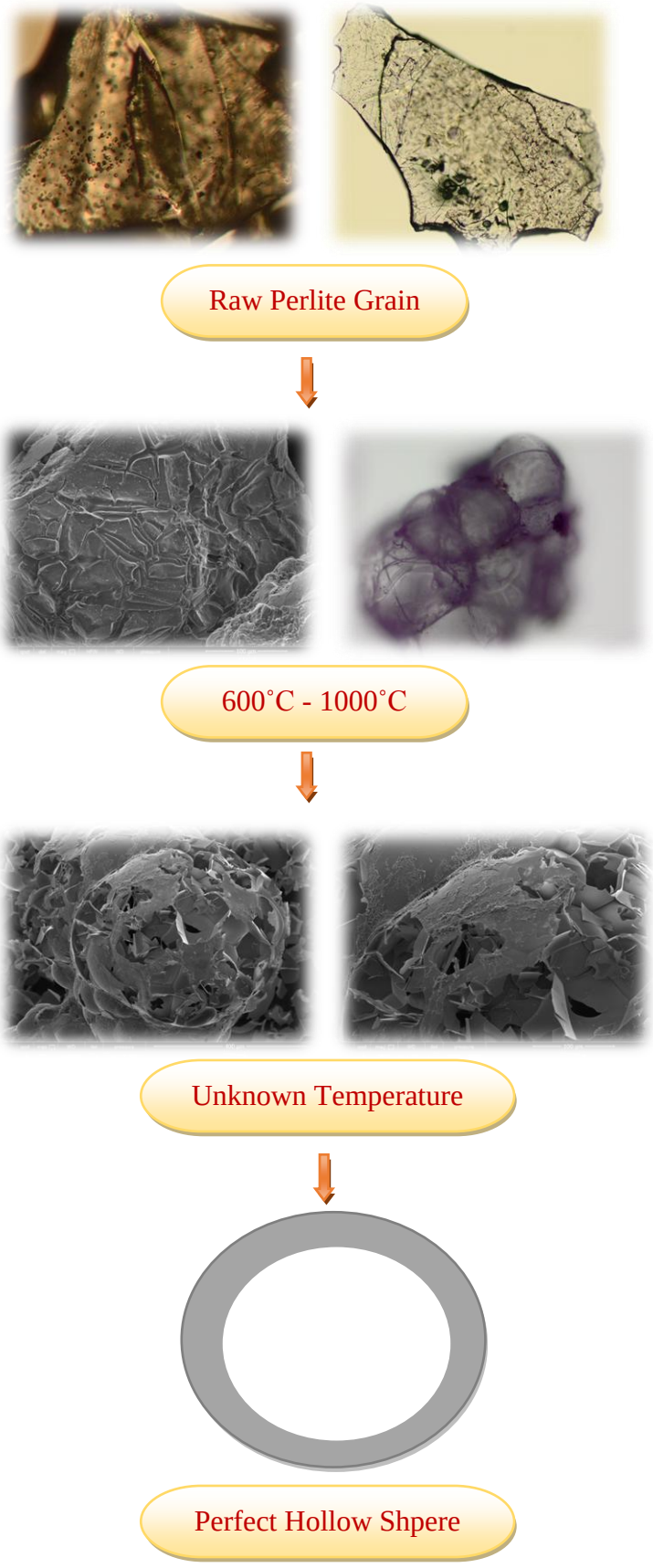


Figure 3.42. Stages of perlite expansion as morphological.

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APPENDIX A.

CHANGE IN BULK DENSITY

In section 3.2.2. were shown the change in bulk volumes for the four perlite samples measured at 600 ˚C, 800 ˚C, 900 ˚C and 1000 ˚C. At the same conditions were arranged for the change in bulk densities and were shown in both table and graphical (Table A.1. and Figures A.1.a-b-c-d.).

Table A.1. Change in Bulk Density for the four perlite samples measured at 600˚C, 800˚C, 900˚C and 1000 ˚C. The results are given for four different size ranges of raw perlite before expansion.

Perlite Source	Heat Treatment Temperature	Particle Size Range (µm)			
		400-500 µm	315-400 µm	200-315 µm	160-200 µm
Bergama 01	600˚C	15%	13%	6%	6%
	800˚C	58%	40%	34%	11%
	900 ˚C	55%	47%	38%	38%
	1000˚C	87%	85%	72%	67%
Bergama 02	600˚C	12%	5%	6%	4%
	800˚C	27%	39%	16%	16%
	900 ˚C	40%	34%	22%	16%
	1000˚C	45%	56%	26%	24%
Avdan	600˚C	1%	2%	3%	0%
	800˚C	36%	28%	24%	29%
	900 ˚C	48%	50%	59%	45%
	1000˚C	80%	62%	74%	80%
Mollatepe	600 ˚C	0%	3%	1%	0%
	800˚C	10%	20%	12%	13%
	900 ˚C	30%	23%	22%	24%
	1000˚C	32%	22%	26%	24%

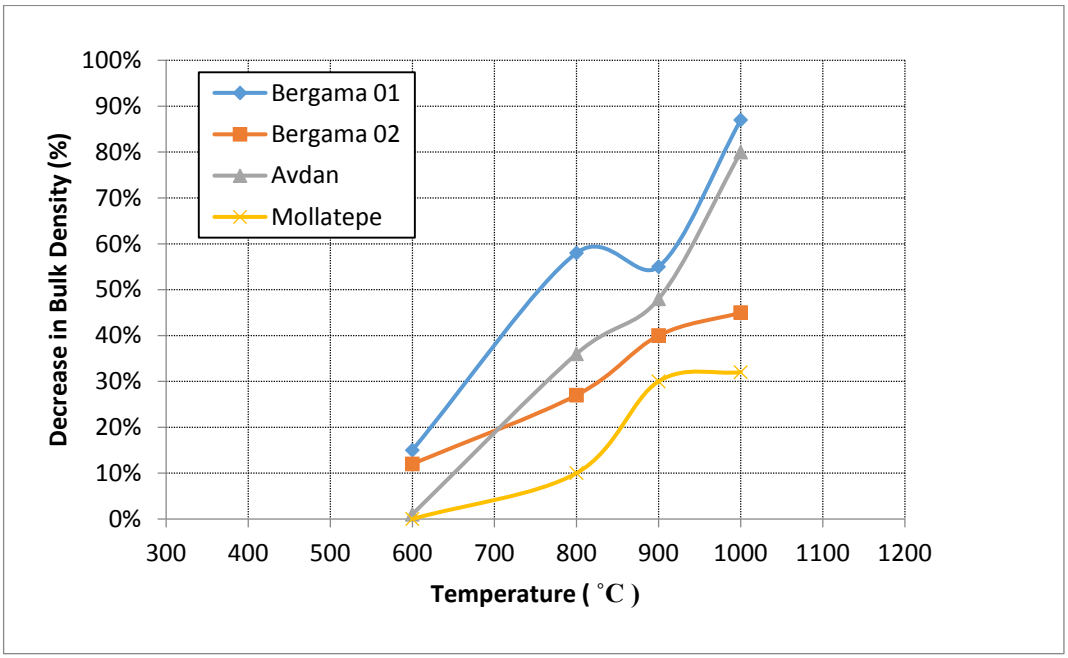

Figure A.1.a. The percentage of decreasing in bulk density with increasing temperature all of the perlite samples at 400-500µm.

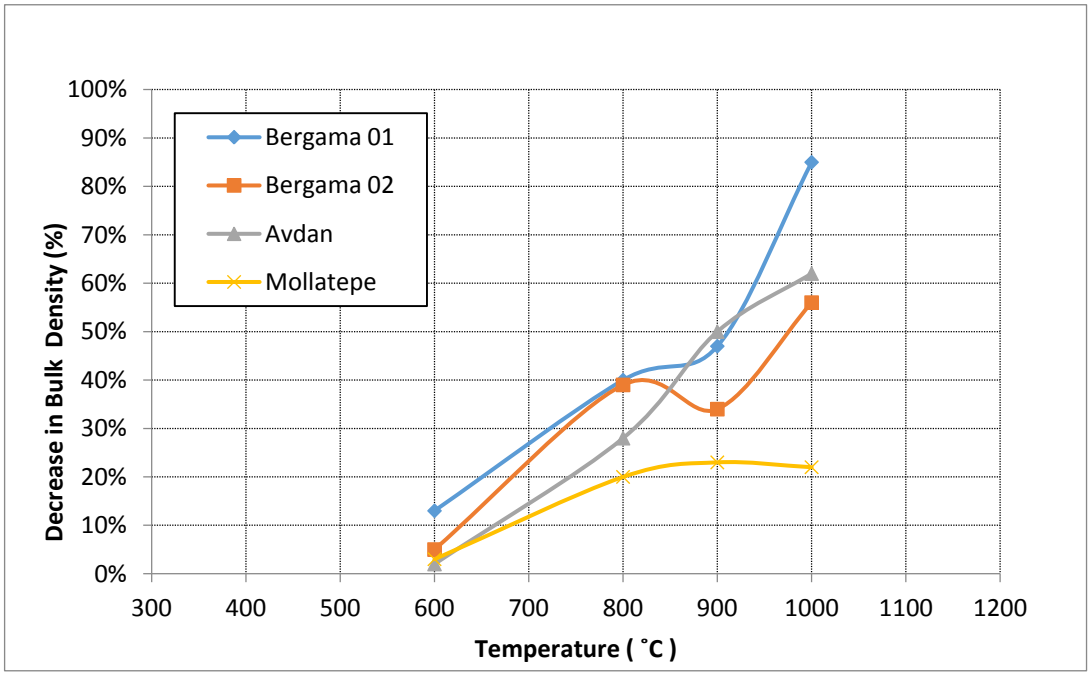

Figure A.1.b. The percentage of decreasing in bulk density with increasing temperature all of the perlite samples at 315-400µm.

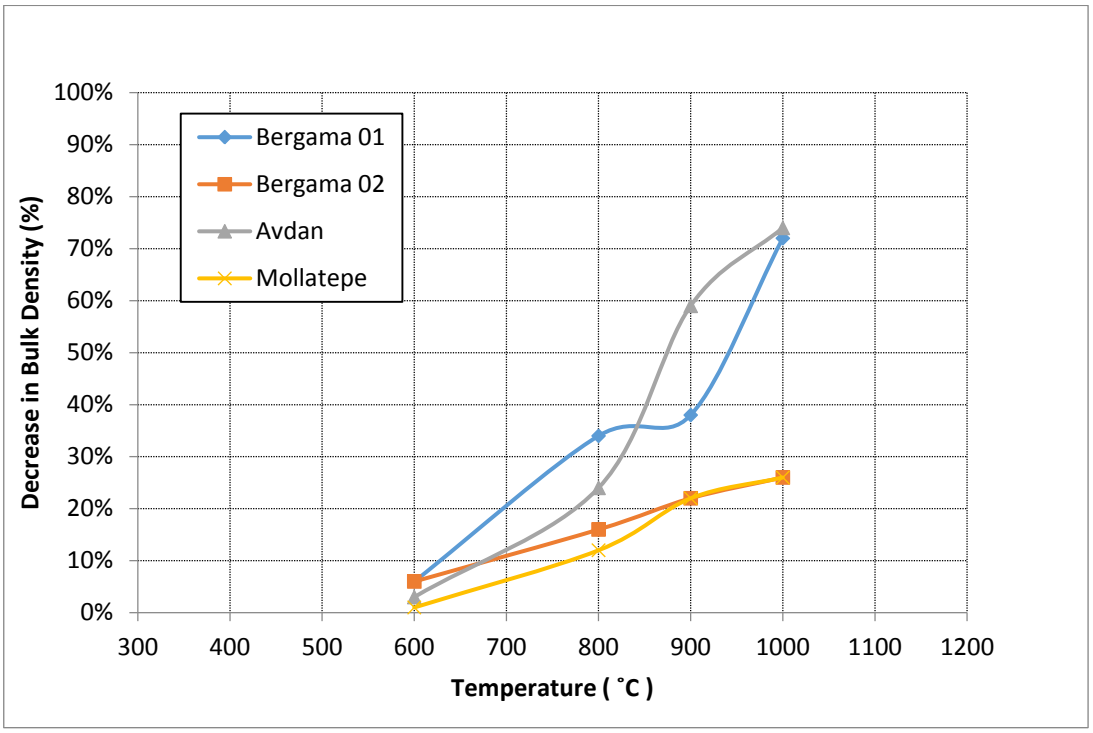

Figure A.1.c. The percentage of decreasing in bulk density with increasing temperature all of the perlite samples at 200-315µm.

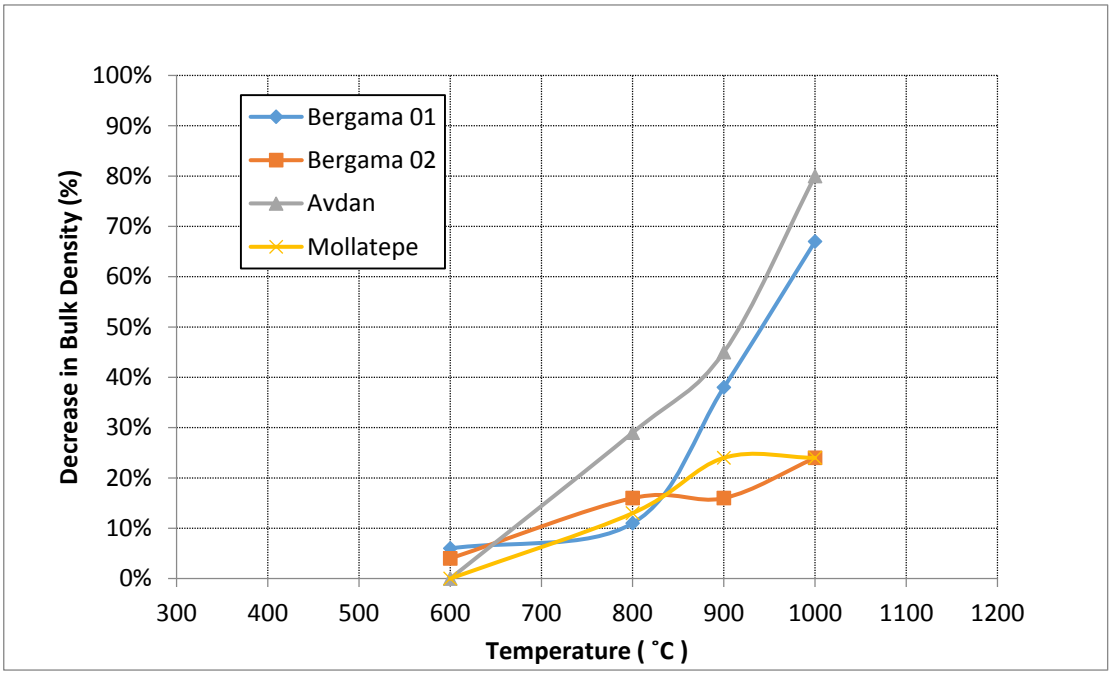

Figure A.1.d. The percentage of decreasing in bulk density with increasing temperature all of the perlite samples at 160-200µm.