FTIR Spectroscopy for Gypsum after Treatment with Steam Pressure

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Abstract

The effect of 2 bar steam pressure was studied on the Mm and Ms mineral sources of calcium sulphate dihydrate $CaSO_4.2H_2O$ with and without milling respectively, which is than compared with autoclave calcium sulphate hemihydrate Mα ; α-hemihydrate, and $CaSO_4.2H_2O$ annular; Ma by using FTIR spectroscopy technique at a wave number range 500-4000 Cm^{-1}. The transmittance ratio; {T*-ratio = [(T660/T1620) x 100] } for Mα, Ma, and Mm, was 96.96 %, 95.26% and 72.47% respectively compared with 82.70% at Mm which is treated with 2 bar steam pressure; MP2. The transmittance ratio; [T*-ratio] decreased in Ms after the steam pressure (1-6 bar) treatment. The effect of time 0.5 to 2.5 h on passing under 6 bar steam pressure on Mm displays the wave numbers at the range 2000 - 2500 Cm^{-1} which nearly reaching the same to spectrum of Mα. FTIR– spectrum at the range 500-1500 Cm^{-1} for AL-ahliyah gypsum (gypsum-Iraqi plaster); Mh which is calcium sulphate dihydrate heated in open vessel and Mm on treatment by 6 bar steam pressure for 2.5 h; MP6 was studies the destruction of this spectrum range comparing with Mα. The disadvantage of steam pressure on gypsum was observed by stretching of OH-group at the rang 3000-4000 Cm^{-1} in Mm, where the increasing of steam pressure in MP2 and MP6 caused in disappearing the wave numbers of 3609 and 3553 Cm^{-1} compared with the wave numbers appearing in Mα and Mh. This study showed the steam pressure may be used as a good parameter to change Mm to Mα which equivalent to the physical properties of dental material.

Introduction

Gypsum is one of the most useful materials in the dental field; non serve the profession more adequately than the products of gypsum (1). Gypsum products are used in dentistry for preparation of study models of oral and maxillofacial structures and as important adjuncts to the dental laboratory operation.

Various types of gypsum used to form molds and casts on which dental prostheses and restorations are constructed (2). From mineral source of gypsum Ms, there are two methods of preparing hemihydrate of gypsum, one of these methods called calcined calcium sulphate by heating in an open vessel 120 C° to perpetrate β–hemihydrate; Mβ and another method is called autoclaved calcium sulphate by dehydration in autoclave under steam pressure at 120–150 C° to perpetrate α-hemihydrate; Mα.

There are two types of hydrates of calcium sulphate, one type is α-hemihydrates and another β-hemihydrates Fig.(1) and the difference between these types are the result of the differences in crystal size, surface area, and the degree of lattice perfection (3).

Mα; α-hemihydrate is much useful more than gypsum since its having a good physical properties (3).There are several study about physical properties of many dental materials such as gypsum (1, 4, 5, 6) α-hemihyrate (1) and others dental material (2, 7) by using different techniques as nuclear magnetic resonance-NMR(8), Raman spectrum (9, 10), k-Edge x-ray absorption (5), neutron powder diffraction (11), x-ray diffraction (12) as wall as spectroscopic techniques, and one of these techniques, infrared-IR spectroscopy which used for CaSO4 at 1968 (13) and used Fourier Transformer Infrared–FTIR technique to measure spectroscopic properties of pigment (14), hemihydrate (7), quantification of $CaSO_4.2H_2O$ mixtures, effect of ultrasound of some dental material (16), formation of γ-CaSO4 phase in dehydration process gypsum (17) and properties of conversion analysis of all-in-one adhesives (18). The steam pressure is one of the methods of preparing α-hemihydrate from gypsum, and many studies took the effect of pressure on stone (4).

So in this study preparation of α-hemihydrate by using autoclaved calcium sulphate of mineral source of gypsum was treated with steam pressure and the effect of treatment was measured by using FTIR spectroscopy, which reflected some physical properties compared with Mα, in addition this study improved the effect of milling on mineral source of gypsum; Ms, and time of passing under autoclaved high steam pressure on gypsum, and comparison was made for the results of FTIR–spectrum of treated gypsum with steam pressure with Mα ; α-hemihyrate, Mh; AL-ahliyah gypsum-Iraqi plaster (equivalent to β-hemihyrate), and Ma ; annular $CaSO_4.2H_2O$.

Material and Methods

One hundred gm of mineral source of calcium sulphate $CaSO_4.2H_2O$; Ms was milled milling with hand mortar to get Mm as fine powder. AL-ahliyah gypsum Mh and α- hemihydrate; Mα and all properties of the above mineral sources of hydrates calcium sulphate (3) were shown in (Fig.(1)) which diagramed the mineral sources of gypsum, calcium sulphate dehydrate $CaSO_4. 2H_2O$; Mm and Ms with and without milling respectively. There are other types of calcium sulphate dihydrate can be produced from other industries such as Ma and Mα; α-hemihydrate (autoclave calcium sulphate hemihydrate) which are produced by heating in autoclave under steam presser 120-130C° and other types is calcium sulphate dehydrate annular-$CaSO_4$ $2H_2O$; Ma.

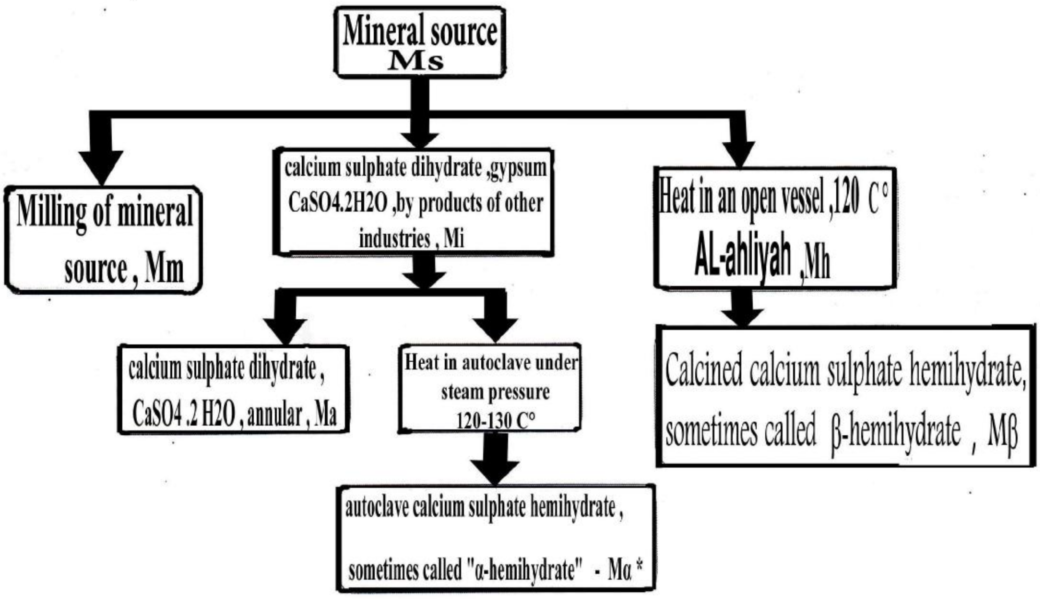

Fig. (1) Diagram of mineral source of hydrates calcium sulphate (3)* : From mark Elito stone with following properties , water / powder ratio 25 ml / 100 gm, working time : 8¢, setting time (vicat) : 14¢, setting expension 2 h : 0.08%, compressive strength 1 h : 42 MPa.

The mount used of above samples Ms, Ma, Mh, and Ma was about 25 gm in all experiments. The steam pressure 2 bar for 1h was used to treated Mm by using autoclave Model-Gnatus-21 liter.

High steam pressure 6 bar with passing time 0.5 to 2.5 h on Mm was treated by using autoclave-Portable Express Equipment 12 liter, after egested their safety valve to reach 6 bar and was conducted at the Department of Basic Science-Dental College. FTIR-spectrum measured for Mm, Ma, Ma, and Mh was done by using FTIR spectrophotometer model BRUKER–TENSOR–27 with KBr pellet, then pellet was transferred into FTIR-spectrophotometer to measure their spectrum at a wave number ranging from 500 to 4000 Cm^{-1}. The wave numbers which were taken into account were 660, 1620 Cm^{-1} and the transmittance ratio [T*-ratio] calculated by the following equation ;

$[T^*\text{-ratio}] = [(T_{660}/T_{1620})\times 100]$ *equation 1*

Where T_{660} and T_{1620} are transmittance percent for samples at wave numbers 660 Cm^{-1} and 1620 Cm^{-1} respectively.

Results

(Fig.(2)) showed FTIR - spectrum at the wave number range 500 - 4000 Cm^{-1} for Mm which shown the wave numbers 600, 652, 1151, 1620, 3548, 3609 Cm^{-1}.

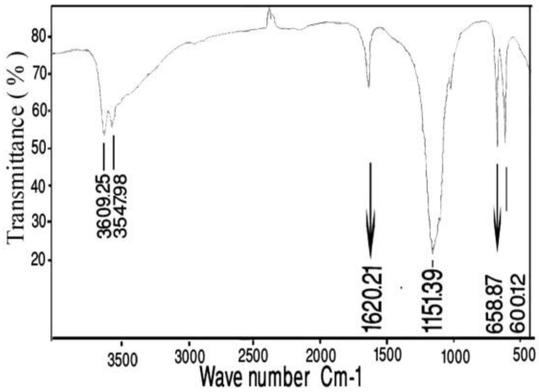

Fig. (2) FTIR-spectrum at range 500-4000 cm^{-1} for Mm : milling of mineral sources of gypsum.

Table (1)
Wave numbers in Cm^{-1} of FTIR spectrum for following mineral source products of dihydrate calcium sulphat $CaSO_4.2H_2O$.

Present study				Mh*
Mm	**Mα**	**Ma**	**Mh**	
600	559	601	600	602
659	663	667	659	669
1006	---	---	1007	1005
---	1094	1124	1094	1117
1151	1153	1620	1152	1145
1620	1621	1686	1620	1621
---	---	2115	---	1685
	---	2236		---
	---	2348		---
	---	3241		---
	---	3398		---
---	---	3489		
3548	3553	---	3554	---
---	---	----	---	3405
3609	3610	---	3610	3547

Mm : milling of mineral source –Ms
Mα : α- hemihydrate
Mh: AL-Ahliyah gypsum
Ma : $CaSO_4$. $2H_2O$, annular
**** : Gypsum – Mh from Passad, et. al. 2005(18).***

as given in (Table (1)) compared with Prasad - study - 2005 (17). In measurement the transmittance percent (transmittance%) for Mm at wave number 660 Cm^{-1} and 1620 Cm^{-1} were found to be 46.7, 64.2 respectively, that means the transmittance ratio [T*-ratio] for Mm was 72.47% (*equation -1*).

(Fig.(3)) showed FTIR - spectrum at the wave number range 500-4000 Cm^{-1} for Mα and their pure wave numbers appeared in 599, 663, 1153, 1621, 3553 and 3610 Cm^{-1} (Table (1)) and the transmittance ratio [T*- ratio] for Mα equal to 96.96% compared with 72.47% of Mm.

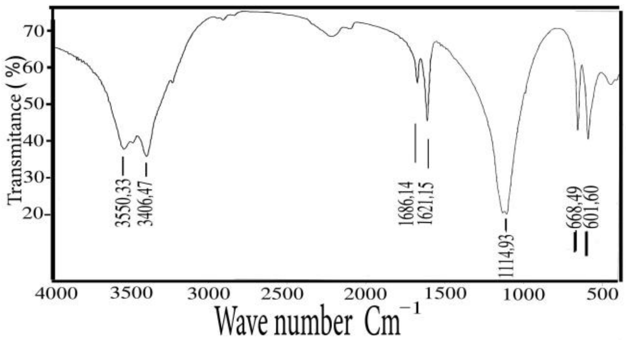

Fig. (4) FTIR at the wave number range 500-4000 cm^{-1} for MP2 : milling of mineral source Mm with 2 bar steam pressure at 1 h.

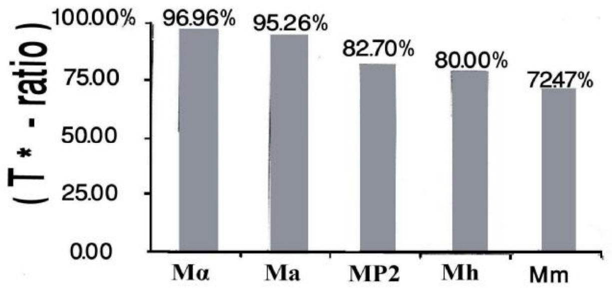

Fig. (5) Transmittance ratio {T*-ratio] from FTIR for Mn, Ma, Mh, MP2 and Ma.
Mm : milling of mineral source of gypum
Mh: AL-Ahliyah gypsum
MP2: Mm treatment with 2 bar steam pressure at 1 h.
Ma : $CaSO_4$. $2H_2O$ annular
Mα : α- hemihydrate

(Fig.(4)) showed FTIR - spectrum at the range 500-4000 Cm^{-1} for MP2; Mm which treatment with 2 bar steam pressure for 1 h.

The [T*-ratio] for MP2 equal 82.70% as shown in (Fig.(5)) which is obtained with another [T*-ratio] for Mm, Mh and Ma as wall as Mα. From (Fig.(5)) T*-ratio for Mα has a greatest value compared with Mm, Mh, MP2 and Ma values, and that [T*-ratio] for MP2 was 82.7% which was nearly compared to 96.96 % of Mα

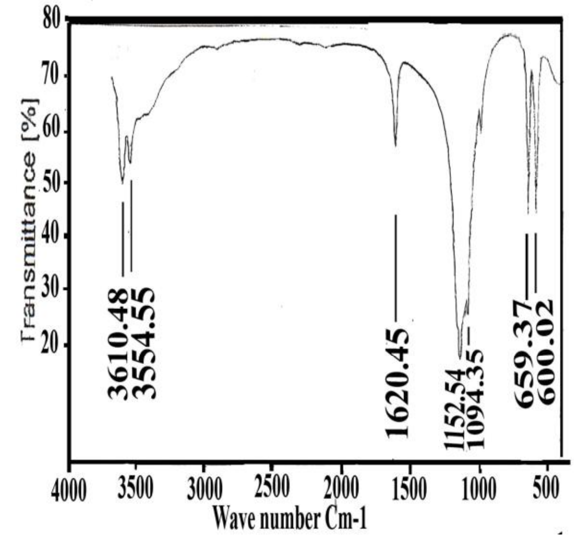

Fig.(6) FTIR-spectrum at the wave number range 500-4000 cm^{-1} for Mh : AL-ahliyah gypsum.

(Fig.(6)) showed FTIR-spectrum at the range 500-4000Cm^{-1} for Mh; AL-ahliyah gypsum (*gypsum-Iraqi plaster*) and their wave number appeared at 600, 659, 1153, 1620, 3555 and 3610 Cm^{-1} (Table (1)).

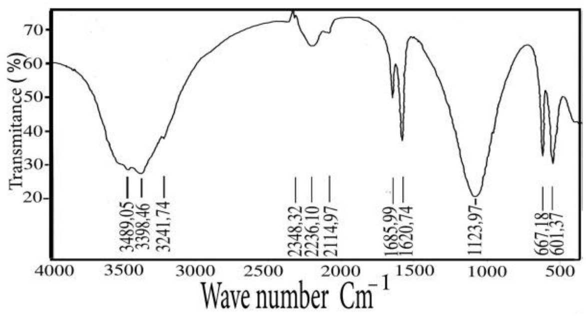

Fig.(7) FTIR-spectrum at the wave number range 500-4000 cm^{-1} for Ma : calcium sulphate dihydrate annular $CaSO_4$. $2H_2O$.

(Fig.(7)) showed the FTIR spectrum at the range 500-4000 Cm^{-1} for Ma annular which reflected one of the gypsum products with wave numbers appeared in 601, 667, 1124, 1620 and 1686 Cm^{-1} where their [T*-ratio] equal to 95.26% (Fig.(5)).

The treatment of Ms with high steam pressure 1-6 bar with passing time 1 h causing a decrease in [T*- ratio] as shown in (Fig.(8))

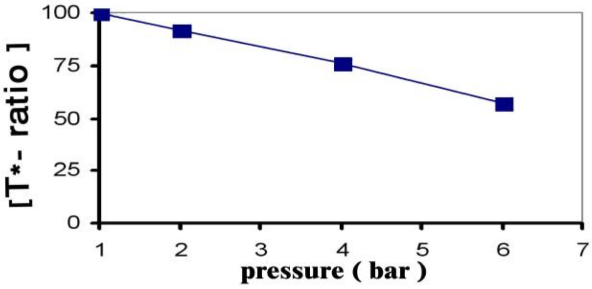

Fig. (8) [T*-ratio] of FTIR spectrum for minerial source, Ms which effected steam pressure at range 1-6 bar during 1 h.

and the minimum value of [T*-ratio] was 57.2% at 6 bar. The effect of time passing on steam pressure on Mm obtained by (Figure-9) which shown FTIR-spectrum at the range 2000-2500 Cm^{-1} for Mm and Mm which treatment 6 bar steam presser at passing time 0.5, 1.5, 2.0 and 2.5 h comparing with Mα.

(Fig.(9)) showed that 2000-2500 Cm^{-1} spectrum for 6 bar steam pressure at 2.5 h was in a good agreement with Mα, although a difference was observed in the wave number at 2400 Cm^{-1}.

The effect of 6 bar steam pressure on gypsum my be shown in by (Fig.(10)) which is obtained from FTIR-spectrum at a wave number ranging from 500-1500 Cm^{-1} for Mh, Mα and MP6; Mm for steam pressure treatment of 6 bar with passing time 2.5 h.

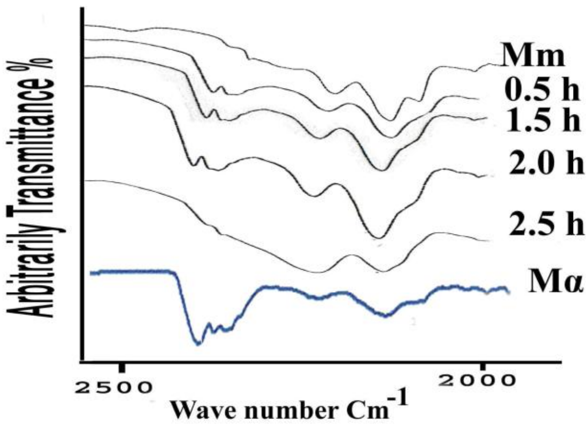

Fig.(9) FTIR-spectrum 2000-2500 cm^{-1} for Mm and Mm which effected by steam pressure 6 bar at 0.5, 1.5, 2.0, and 2.5 h compared with Ma.

From (Fig.(10)) the effect of steam pressure in MP6 was different through disploing of their spectrum comparing with

Mα. When measuring the stretching of OH-group gypsum by measuring FTIR spectrum at the range 3000-4000 Cm^{-1} as there in Marcin Antonin Paraizea et. al., 2004 (19) for Mα, Mm, Mh, MP2, MP6 and Ma was observed the disappear of 3609, 3553 Cm^{-1} wave numbers in above samples comparing with Mα and Mm as shown in (Fig.(11)).

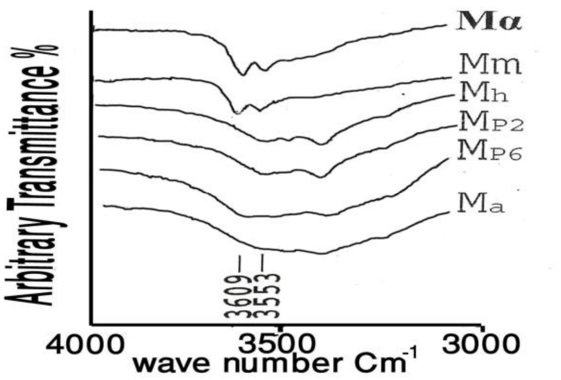

Fig.(10) FTIR-spectrum at the wave range 500-1500 cm^{-1} for M, Mα and MP6; Mm which effected 6 bar steam pressure 25 h.

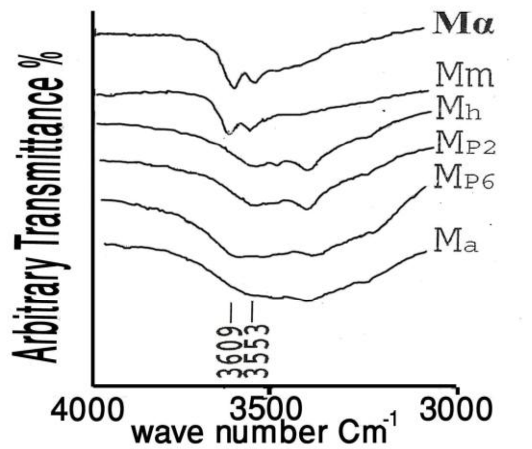

Fig.(11) FTIR spectrum at the range 3000-4000 Cm^{-1} for

Mα : α- hemihydrate
Mm : milling of mineral source
Mh: AL-Ahliyah gypsum
MP2: Mm which effected treatment with 2 bar steam pressure for 1 h.
MP6 : Mm which treatment by 2 bar steam pressure for 2.5 h.
Ma : $CaSO_4 . 2H_2O$ annular.

Discussions

The wave numbers of Mm, Mα and Mh which appear at the range 500- 4000 Cm^{-1} have nearly the same wave numbers as shown in (Table (1)) which are obtained from the present study for Mh and was in a good agreement with wave numbers of gypsum from study of Prasad, et. al. (18). Thus the wave numbers 660, 1620 Cm^{-1} for Mα were more pronounced, so taken into account to calculate the transmittance ratio [T*- ratio] in equation-1.

The total wave numbers appeared in Ma was more than total wave numbers in Mm, Mα and Mh (Table (1)) since this depends on the chemical procedure of production of $CaSO_4.2H_2O$ annular and depend on addition of organic acids (15). The change of wave numbers 660, 1620 Cm^{-1} in Mα compared with Mm was observed in (Fig.(2)) and (Fig.(3)) for Mm and Mα with [T*-ratio] equal to 72.47 % and 96.96% respectively. The transmittance percent at 663 and 1621 Cm^{-1} which equivalent to 660, 1620 Cm^{-1} for Mα was shown in (Fig.(3)). The [T*-ratio] for MP2 decreased to 82.7 % which nearly to 96.96 % the value of Mα as showed in (Fig.(5)), which induced that the treatment of steam pressure on Mm was good parameter to change Mm to Mα, also the effect of heating in an open vessel 120 C^o at Mh also increases the [T*-ratio] to 80.0% nearly to 96.96% of Mα as shown in (Fig.(*6)), but the same in shape of FTIR-spectrum at the range 500-4000 Cm^{-1} of MP2 (Fig.(4)) with Mα (Fig.(3)) was in a good agreement more than Mh (Fig.(6)), although [T*- ratio] of Mh was equivalent to [T*-ratio] of MP2. The effect of the direct heating in Mh compared with Ma reflected by the different in [T*-ratio] values of 80.0%, 95.26% for Mh and Ma respectively which obtained in (Fig.(6)) (Fig.(7)). The effect of increasing 1-6 bar steam pressure at 1 h on Ms decreased the [T*-ratio] from 100% to 57.2% at 6 bar, that refer to the effect of steam pressure on mineral source of gypsum without milling cases the disadvantage properties as a result of decreasing of [T*-ratio], as wall as that my be retuned to morphological of crystal structure of $CaSO_4$ (20).

Increasing of time from 0.5 to 2.5h which passing under 6 bar steam pressure on Mm was change at the wave range 2000-2500 Cm^{-1} to seemly spectrum for Mα as shown in (Fig.(9)). But there is difference at the wave number range 2400 Cm^{-1} which means there is chemical additive with steam pressure used to production Mα. The effect of high steam pressure 6 bar by MP6 was cased disadvantage effect comparing with Mα and Mh which obtained in the wave numbers range 500-1500 Cm^{-1} spectrum as shown in (Fig.(10)).

FTIR-spectrum at the range 3000-4000 Cm^{-1} disappeared wave numbers which appears in Mα and Mm at 3609 and 3553 Cm^{-1} comparing with disappearing of these wave numbers in the Mh, MP2, MP6 and Ma as showed in (Fig.(11)) which describe that Mα and Mm having the same spectrum at the range 3000-4000 Cm^{-1} and reflected no change in OH-group (17, 18). From above figure stretching of OH - group disappearing gradually for Mh, MP2, MP6 and Ma. The effect of steam pressure and chemicals addition production of Ma which obtained for production of Ma and may be included using together for change Mm to Mα and having acceptable physical properties of dental materials. The stretching of OH-group which obtained from Mh, MP2, MP6 compared with Mα and Mm might be high sensitive technique as wall as using another techniques which depended on crystal structure with morphology of $CaSO_4$ (21) and reflected the bounding of H_2O molecules in CaSO4. $2H_2O$ (13, 21). It is demonstrated in this study that the affect of steam pressure on mineral source of gypsum study may be used with the void size and quantity in dental stone (22), so the evaluation of any treatment with gypsum take into account the preservative of OH-group.

And to preparation Mα my be using chemical additive materials (23) with steam pressure and matching with standard mechanical properties of Mα.

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الخلاصة

تم دراسة تأثير ضغط البخار (2 بار) على Mm و Ms المصادر الخام لمادة الجبس المتكون من كبريتات الكالسيوم اللامائية –$CaSO_4$. 2H_2O مع وبدون الطحن على التوالي، وتم مقارنتها مع Mα (α-hemihydrate) و Ma: كبريتات الكالسيوم النقية المختبرية باستخدام تقنية مطيافية تتحول فورير تحت الحمراء–FTIR عند مدى العدد ألموجي $500–4000\ Cm^{-1}$. كانت نسبة النفاذية المئوية لطيف FTIR المعطى بالمعادلة

$$T^*\text{-ratio} = [(T_{660}/T_{1680}) \times 100]$$

لكلاً من Mα Ma , Mm تساوي 96.96%, 95.26% 72.74% على التوالي، مقارنة بـ 82.70% عند Mm الذي تم معالجته بضغط بخار 2 بار والموسوم بـ MP2. تبين أن [T*-ratio] تتناقص عند Ms بعد معالجتها بضغط بخار 1– 6 بار، وان تأثير الزمن المار لتأثير ضغط البخار من 0.5 الى 2.5 ساعة تحت ضغط بخار 6 بار على Mm يؤدي إلى اختفاء الأعداد الموجية عند المدى الموجي ($2000 – 2500\ Cm^{-1}$) إلى حد إن يصل إلى نفس سيماء الطيف عند Mα. تم دراسة تشتت طيف -FTIR عند

المدى $500-1500 Cm^{-1}$ لجبس الأهلية Mh : Al-Ahlyiah (كبريتات لكالسيوم اللامائية المحضرة بطريقة التسخين بوعاء مفتوح – جبس منتج عراقي) و MP6 التي هي عبارة عن Mm معالج بضغط بخار 6 بار عند 2.5 ساعة و مقارنة ذلك مع Mα. تبين أن مساوئ استخدام ضغط البخار على مادة الجبس هي بملاحظة ظهور أمتداد لمجاميع OH عند المدى ($3000 - 4000 Cm^{-1}$) في حالة Mm، بينما أدت زيادة ضغط البخار عند MP2 و MP6 إلى اختفاء الأعداد الموجية $3553 Cm^{-1}$، $3609 Cm^{-1}$ مقارنة بظهورها عند Mα، Mh.

بينت هذه الدراسة إمكانية استخدام ضغط البخار كعامل جيد في تحويل المصادر الخام لمادة الجبس Mm الى Mα : α-hemihydrate الذي يمتلك المواصفات الفيزيائية الملائمة في مجال مواد طب الأسنان.