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Evaluation of some mechanical properties of type II dental plaster after addition of Halloysite nanoclay.

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ABSTRACTObjectives:the study evaluates the effect of adding halloysite nanoclay on some properties (initial setting time, linear setting expansion and compressive strength) of type II dental plaster.

Materials and Methods:type II dental plaster (BDH- England) is used in this study. Initial setting time, linear setting expansion, and compressive strength of dental plaster that is incorporated with different concentrations (0.5%, 1%, and 2%) of Halloysite nanoclay are evaluated and compare the results with results of unmodified specimens which called control specimen. The statistical analysis of these results is done by mean, standard deviation, ANOVA and duncan's multiple range test. These results were significant in $p \leq 0.05$).

Results:the results reveal that the inclusion of halloysite nanoclay change the evaluated properties of dental plaster. halloysite nanoclay additives affect initial setting time, linear setting expansion, and compressive strength of dental plaster so initial setting time was decreased significantly when the concentrations of added nanoparticles are increased. The compressive strength of plaster is increased significantly when the concentration of added halloysite nanoclay is increased and the linear setting expansion is significantly decreased in comparison to control specimens.

Conclusion: from the results of this study we can conclude that incorporation of halloysite nanoclay in dental plaster can result in significant improvement in the mechanical properties of dental plaster.

Keywords:halloysite nanoclay, Initial setting time, linear setting expansion, compressive strength

I. INTRODUCTION

Dental Plaster is the first gypsum dental product accessible in dentistry. It's made by finely crushing gypsum rock and then heating it in an open container. This direct and quick heating in the open air shatters the crystal by removing some of the crystallisation water. The resulting powder is porous and uneven in shape. The cheapest and weakest of the three gypsum products is plaster. It's mostly used for preliminary casts for complete dentures and attaching casts to a mechanical device called an articulator, where strength isn't a big deal. Plaster is mostly white in its colour and is also known as a beta-hemihydrate or as type II plaster. In the past time, plaster is modified by chemical process to be used as material for impressions making and is referred to as an impression plaster [1]. Dental plaster is, the material that contain beta-hemihydrate particles, utilized for study models, a material for set-up of the working models upon the articulators, and as a dental lab material. In orthodontics, the white gypsum that contains β-hemihydrate is utilized as a study models to give a three-dimensional image of occlusion of the patient, by this way, it is easier to decide treatment options by the dentist [2].Many studies have shown that adding various substances to the powder of calcium sulphate hemihydrate or the gauging water affect on the setting time and expansion during setting and the incorporation of chemical materials to gypsum products alters not only the physical characteristics, but also the morphology of the crystals [3]. Halloysite is an aluminosilicate clay mineral with the empirical formula al2si2o5(oh)4. Its main constituents are oxygen (55.78%), silicon (21.76%), aluminium (20.90%), and hydrogen (1.56%) [4]Nanomaterials are materials with at least one nanoscale dimension (1–100 nm) or their basic units are at this range of the three dimensional space [5]. The effects of incorporation of halloysite nanoclay on the mechanical properties of type II dental plaster will be assessed in this study.

II. MATERIALS AND MEHODS

Three types of measurements were done in this study as following

- Measurements of initial setting time.
- Measurements of linear setting expansion.
- Measurements of compressive strength.

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A total of 20 specimens were prepared for each one of these measurements. the specimens were divided into 4 groups as following:

- Control group: without addition of halloysite nanoclay (5 specimens).
- Group A1: 0,5% weight concentration of halloysite nanoclay (5 specimens).
- Group A2: 1.0% weight concentration of halloysite nanoclay (5 specimens).
- Group A3: 2.0% weight concentration of halloysite nanoclay (5 specimens).

So we have 60 specimens in total for all measurements.

Measurement of initial setting time: The initial setting time is evaluated in accordance to iso 6873 (2013) by use of standard vicate apparatus as showed in figure (1). Vicate apparatus consists of frame carrying a rod, the rod is mobile in up and down direction. The mobile rod weight is 300 gm. Needle with (5 cm) in length and (1 mm) diameter was fixed at one end of the mobile rod. A mold placed on the table of the vacate apparatus which is filled with the mixture of the test specimen.

120 gram of dried dental plaster is mixed with the distilled water in accordance to water \ powder ratio of manufacturer. That will result in a mixture sufficient to fill the molud. The mixture of specimens which prepared to be tested is poured in the mould. The mould is placed on a plate, this plate is made from glass. The molud is filled with the dental plaster mixture and then is wiped with spatula to be leveled with the top of the molud. Then the needle is elevated and the mold is moved to position beneath the needle and 4 mm away from the mould wall at least.

The time required for the setting of each specimen is evaluated by lowering the device needle till it contacted the specimen surface and then adjust penetrometer scale to zero reading and lock the scale on that position with the locking screw. Then the needle is released in order to penetrate the specimen. The penetration is done at each 15 seconds intervals starting 1 or 2 minutes before the anticipated setting time that is determined by the plaster manufacturer.Before each penetration, the needle is cleaned carefully and the specimen is moved to let the new penetration in the specimen to be done. The new area of penetration should be 4 millimeters at least away from the mould wall and from the previous area of penetration. The whole time elapsed from the beginning of mixing procedure to the time when the needle tip firstly unable to penetrate the prepared plaster specimen to a 2mm depth is recorded as the vicat or initial setting time for the specimen.

Figure (1) initial setting time measurements.

Measurements of linear setting expansion: Measurements of linear setting expansions are done according to ISO specification number 6873 (2013).

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The device consists of 104 mm length and 58 mm width trough that have fixed end and one mobile slide end on the other side. A dial gauge is connected to the mobile slide end and used to determine the amount of movement of the mobile slide. The dial gauge should be arranged on zero before pouring the mixture. The trough must be painted by separating medium. Figure (2). Mixing procedure is done by using 120 gm of the dental plaster powder with distilled water according to the water/ powder ratio determined by manufacturer. This procedure would result in a creamy mixture adequate to fulfill the trough.The dial gauge is on zero reading and the mixture is poured into the trough. The alteration in the dial gauge readings during setting of specimen indicate that changes in specimen length are occurred. First reading is taken (60 ± 1) second before the initial setting time that previously measured and then the final reading is taken 2 hours after mixing.Calculation of setting expansion is done by the use of following formula:

$$\text{Setting expansion } \% = \frac{FR-IR}{L}\times 100$$

(Salem et al., 1997)

(fr): the final reading of the device gauge which is recorded exactly after two hours from the starting of mixing procedure.

(ir): the initial reading of the device gauge which is recorded one minute before the initial setting time.

(l): the total length of plaster specimen which is measured by the digital scale.

Figure (2) linear setting expansion.

Measurements of compressive strength: Measurements of compressive strength are done according to ISO specification number 6873 (2013).Cylindrical samples 40 mm length and 20 mm in diameter is made in a split mold figure (3), 5 cylindrical specimens are prepared for each test groups. Mixing procedure is done by use of 220 gm of the dental plaster powder with distilled water according to the water/ powder ratio determined by manufacturer.Prior the pouring of the mixture, the mold is coated with a layer of separating medium in order to facilitate specimen removal from the mold after setting. Then the prepared mixture is poured inside the molud. The mould was fixed on a plate of glass. Vibrator is used to vibrate the mould during pouring for approximately 30 seconds in order to decrease air bubbles formation.

Figure (3) split mold

The over filled mold must be covered with another glass plate and pushed firmly against the top surface of the mold. Specimens are removed from the split mold after half hour from the

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beginning of mixing procedure and stored in air at 50±10% relative humidity and 23c°± 2c°. One hour after the start of mixing, specimens are crushed by load applied to the both ends of each specimen by compressive strength testing machine (tinus olsen ltd, h50kt, USA) till the specimen crushed. Figure (4). The setting criteria of compressive strength testing machine is (load 5000 n, extension 1000 mm, speed 0.5mm/min). Compressive strength testing machine is attached to computer. A special software is used to process the results and the result for each specimen is printed on paper. The testing machine produces a curve that represents specimen crushing steps and contain the maximum load at which the specimen was crushed (figure 5). The maximum load that causes crushing of the specimen is used to calculate the compressive strength by using the following formula:

Cs = f ÷ a

Where cs is the compressive strength in (megapascal), f is the maximum load causes crushing of the specimen in (newton) and a is the cross sectional surface area in (mm).

figure (4) compressive strength testing machine

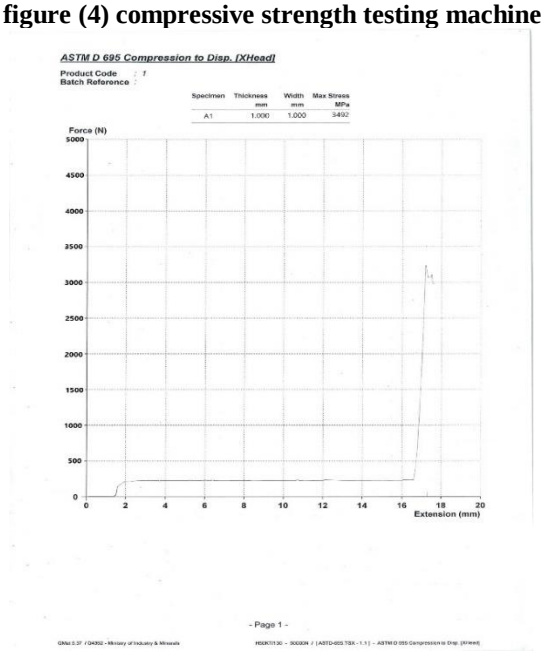

figure (5) compressive strength measurement.

Statistical Analysis:A software program was used to perform statistical analysis (IBM SPSSversion 22). The results of the readings were statistically examined by using (One Way-ANOVA Test) was

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used to identify the existence or absence of a significant difference between groups, at the 0.05 level of significance, and to establish the significant difference between the groups, Duncan's Multiple Range-Test was performed.

III. RESULTS

Initial setting time results: Figure (6) shows the mean and standard deviation values of initial setting time of dental plaster that is incorporated with different concentrations of halloysite nanoclay ($al_2si_2o_5(oh)_4$).One-way analysis of variance (ANOVA) shown in table (1), which is used for control group and for other groups (0.5%,1.0%, and 2.0%) of $al_2si_2o_5(oh)_4$ nanoparticles additives shows that the initial setting time values of dental plaster related to different concentration of $al_2si_2o_5(oh)_4$ nanoparticles is significantly different at $p \leq 0.05$.Initial setting time is decreased significantly when the concentration of $al_2si_2o_5(oh)_4$ nanoparticles additives increased.

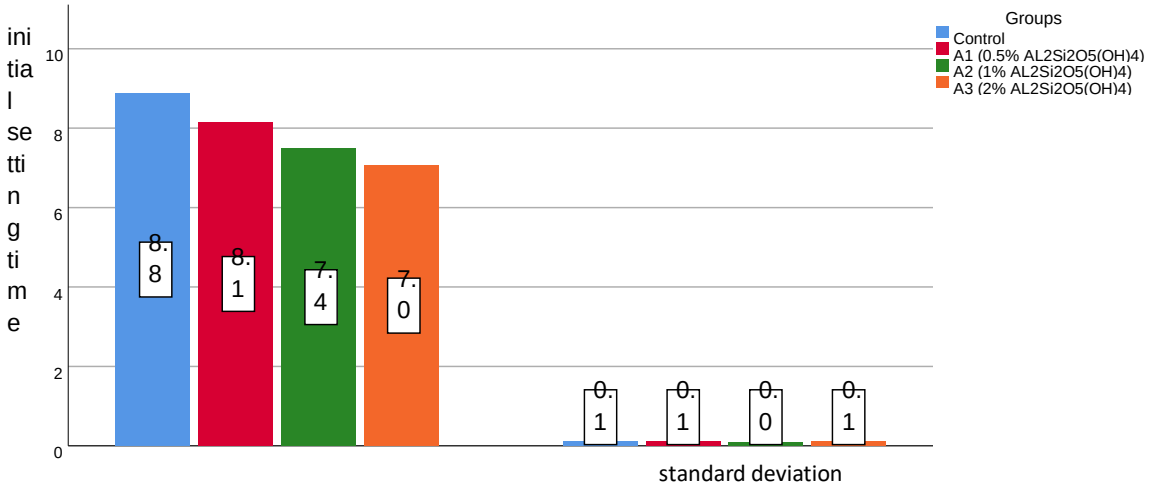

Figure (6) mean andstandard deviation of initial setting timevalues of different concentrations of al2si2o5(oh)4 nanoparticles.

Table (1) ANOVA for initial setting time comparison related to different concentration of halloysite nanoclay.

S.o.v	Sum of squares	Df	Mean square	F	Sig.
Between groups	**9.492**	**3**	**3.164**	**246.355**	**.000**
Within groups	**.205**	**16**	**.013**		
Total	**9.698**	**19**			

S.o.v: source of variance; df: degree of freedom; f: f value.; sig: significance

Table (2) duncan's multiple range test for initial setting time of different concentrations of halloysite nanoclay.

	Groups	N	1	2	3	4
Duncan	**A3 (2% al2si2o5(oh)4)**	**5**	**7.05980**			

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	A2 (1% al2si2o5(oh)4)	**5**		**7.48940**		
	A1 (0.5% al2si2o5(oh)4)	**5**			**8.15300**	
	Control	**5**				**8.87980**
	Sig.		**1.000**	**1.000**	**1.000**	**1.000**

N: number of sample.

linear setting expansion results: Figure (7) shows the mean and standard deviation values of linear setting expansion of dental plaster that is incorporated with different concentrations of halloysite nanoclay ($al_2si_2o_5(oh)_4$).One-way analysis of variance (ANOVA) shown in table (3), which is used for control group and for other groups (0.5%,1.0%, and 2.0%) of halloysite nanoclay additives shows that the linear setting expansion values of dental plaster related to different concentrations of halloysite nanoclayadditives is significantly different at $p \leq 0.05$.Linear setting expansion is decreased significantly when the concentration of halloysite nanoclay additives increased.

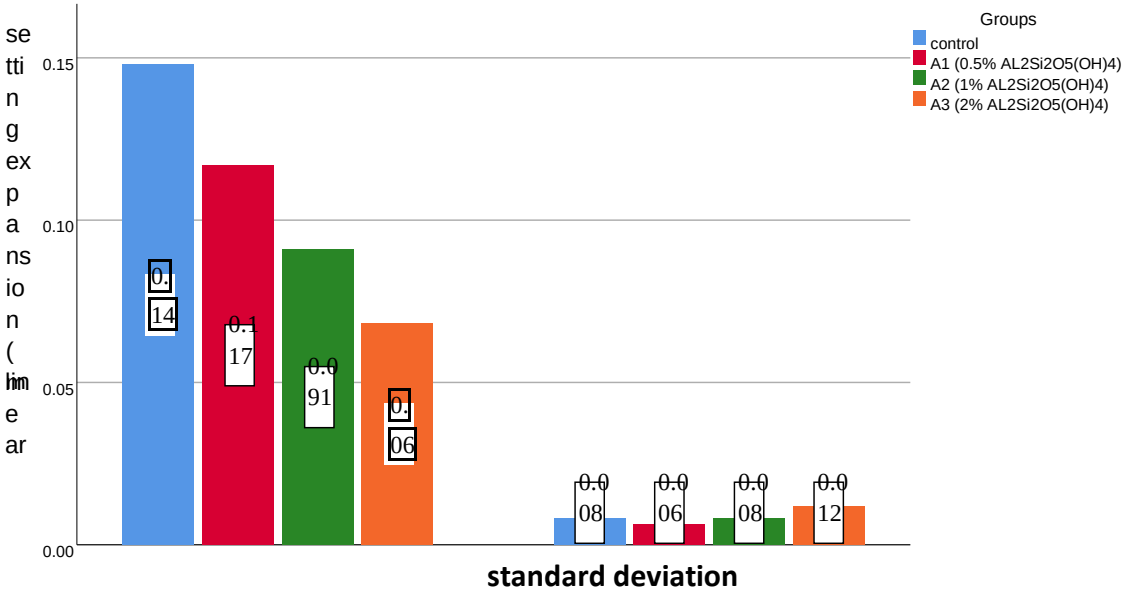

Figure (7) mean andstandard deviation of linear setting expansion values of different concentrations of al2si2o5(oh)4 nanoparticles.

Table (3) ANOVA for linear setting expansion comparison related to different concentration of halloysite nanoclay

S.o.v	**Sum of squares**	**Df**	**Mean square**	**F**	**Sig.**
Between groups	**.018**	**3**	**.006**	**75.669**	**.000**
Within groups	**.001**	**16**	**.000**		

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Total	**.019**	**19**			

S.o.v: source of variance; df: degree of freedom; f: f value.; sig: significance.

Table (4) duncan's multiple range test for linear setting expansion of different concentrations of ahlloysite nanoclay

	Groups	N	1	2	3	4
Duncan	**A3 (2% al2si2o5(oh)4)**	**5**	**.06820**			
	A2 (1% al2si2o5(oh)4)	**5**		**.09100**		
	A1 (0.5% al2si2o5(oh)4)	**5**			**.11680**	
	Control	**5**				**.14800**
	Sig.		**1.000**	**1.000**	**1.000**	**1.000**

N: number of sample.

compressive strength results: Figure (8) shows the mean and standard deviation values of compressive strength of dental plaster that is incorporated with different concentrations of halloysite nanoclay ($al_2si_2o_5(oh)_4$).

One-way analysis of variance (ANOVA) shown in table (5), which is used for control group and for other groups (0.5%,1.0%, and 2.0%) of halloysite nanoclay additives shows that the compressive strength values of dental plaster related to different concentrations of halloysite nanoclay additives is significantly different at $p \leq 0.05$.Compressive strength is decreased significantly when the concentration of $al_2si_2o_5(oh)_4$ nanoparticles additives increased.

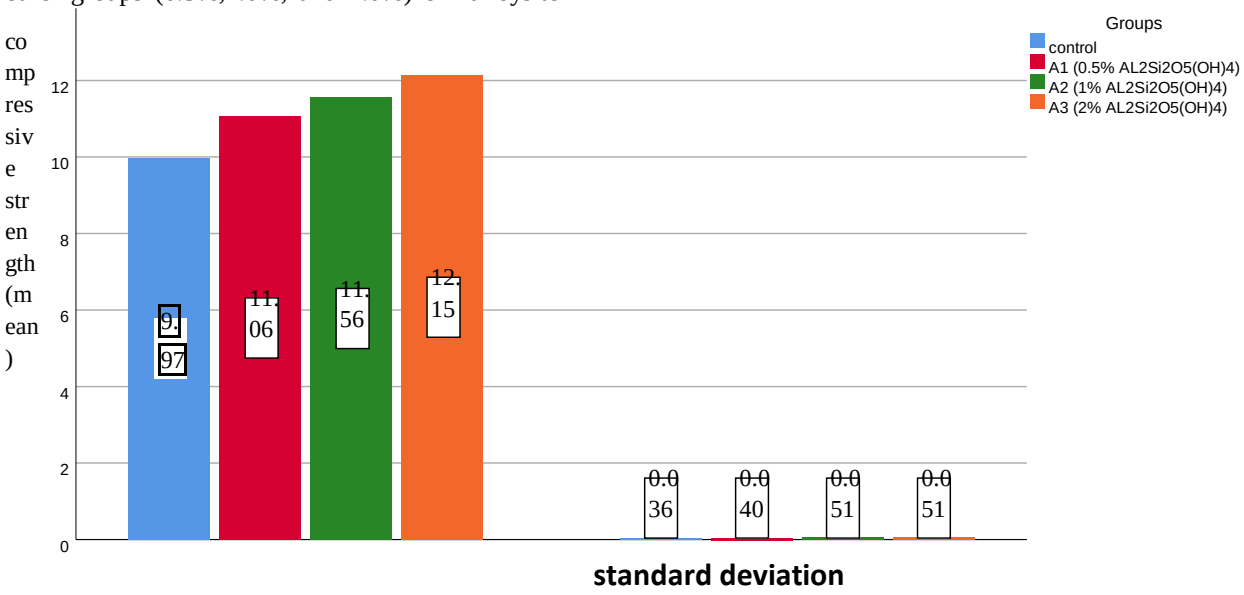

Figure (8) mean andstandard deviation of compressive strength values of different concentrations of al2si2o5(oh)4 nanoparticles.

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Table (5) ANOVA for compressive strength comparison related to different concentration of $al_2si_2o_5(oh)_4$.

S.o.v	Sum of squares	Df	Mean square	F	Sig.
Between groups	12.690	3	4.230	2112.336	.000
Within groups	.032	16	.002		
Total	12.722	19			

S.o.v: source of variance; df: degree of freedom; f: f value.; sig: significance.

Table (6) duncan's multiple range test for compressive strength of different concentrations of halloysite nanoclay

	Groups	N	1	2	3	4
Duncan	Control	5	9.9760			
	A1 (0.5% al2si2o5(oh)4)	5		11.0620		
	A2 (1% al2si2o5(oh)4)	5			11.5560	
	A3 (2% al2si2o5(oh)4)	5				12.1460
	Sig.		1.000	1.000	1.000	1.000

N: number of sample.

IV. DISCUSSION

initial setting time: In this study, we notice that when the concentration of nanoparticles additives increased, the initial setting time is dropped significantly. The results show that adding halloysite nanoclay to plasters reduce the required time for them to set. Because these chemicals function as emulsifiers and reducing the amount of available water in the media, the thickened mixtures will set faster. This is in line with Taqa, a. A., et al. (2015) [6], Another possible cause of the decrease in the initial setting time is because the fact that nanoparticles additives may hasten the chemical changing of the hemihydrate crystals of plaster to the dihydratete, resulting in a reduction in the initial setting time. This agree with (Criage ,2012) [7]

linear setting expansion: The linear setting expansion of type II model plaster should be between the values indicated in ISO specification no. 6873 (2013) for gypsum products. This value range is (0.06-0.30 percent).

Many variables influenced the linear setting expansion related to dental plaster during the transition from the calcium sulphate hemihydrate to calcium sulphate dihydrate and these variations can have a significant impact on cast accuracy and occlusion. (heshmati et al. 2002) [8].

The setting expansion of gypsum is thought to be the result of a mix of expansive forces caused by the growth of gypsum crystals and restrictive forces such as macroscopic exterior restriction and internal restriction caused by the water film's surface tension. The creation of thousands of dihydrate crystals that collide throughout their growth, causes an expansion of the whole mass, that is among the causes that produce the apparent increase in volume (expansion) of gypsum products. (lodovici et al., 2005) [9].

The addition of additives to dental plaster reduces the setting expansion of the resulting specimens, according to the findings. This is due to two factors: first, it is assumed that the spaces between nuclei of crystallisation are smaller after the addition of nanoparticles, resulting in less dihydrate crystal growth interaction and less outward thrust. (Anusavice, 1996) [10].

Secondly, additives that slow or speed up the setting reaction change the hemihydrate crystalline form to a flat tabular shape rather than the spheruletic shape it normally has. This resulted in less crystal interference during growth and, as a result, a lower setting expansion, which is consistent with Wise's findings (2001) [11].

Compressive strength: In the current investigation, the compressive strength value increases as the nanoparticles concentration is increased.Results in this study agree with Hamdy, t. M., et al. (2020) [12] who find that the increase in compressive strength readings of dental plaster when increasing al2o3 nanoparticles concentration.

An increase in the compressive strength of the modified plaster specimens which compared to the control specimens of dental plaster with the additives can be associated with the increase in inter crystallization cohesion among gypsum crystals due to increase in the quantity of gypsum crystals that caused by the increase in the concentrations of additives into plaster and such material may fill the gaps between the crystals during crystal formation, resulting in the production of denser mass. (Khalaf and Mohammed. 2014) [13].

V. CONCLUSION

Conclusions: Within limitation of current study which is conducted in vitro, we can conclude that the incorporation of halloysite nanoclay in plaster can result in significant improvement in the mechanical properties of dental plaster.

halloysite nanoclay additives affect initial setting time, linear setting expansion, and compressive strength of dental plaster so initial setting time is decreased significantly when halloysite nanoclay additives concentrations are increased. Compressive strength of plaster is increased and enhanced significantly when the concentrations of halloysite nanoclay additives are increased and the linear setting expansion is significantly decreased in comparison to control specimens

REFERENCES

[1] Gladwin, M. and M. Bagby (2009). "Clinical Aspects of Dental Materials: Theory." Practice, and Cases2017: Wolters Kluwer Health.

[2] Araújo, T. M. d., Fonseca, L. M., Caldas, L. D., & Costa-Pinto, R. A. (2012). Preparation and evaluation of orthodontic setup. Dental Press Journal of Orthodontics, 17(3), 146-165.

[3] Rashad MM, Mahmoud MH, Ibrahim IA, Abdel-Aal (2004) Crystallization of calcium sulfate dihydrate under simulated conditions of phosphoric acid production in the presence of aluminum and magnesium ions. J Crys Grow.; 267(1-2): 372-379.

[4] Kerr, Paul F. (1992). "Formation and occurrence of clay minerals". Clays and Clay Minerals. 1 (1): 19–32.

[5] Knetsch MLW, Koole LH. (2011) New strategies in the development of antimicrobial coatings: the example of increasing usage of silver and silver nanoparticles. Polymers Basel. 3:340–366.

[6] Taqa Amer A, Nada Z. M. and Tariq Y. K. Bash, (2015) The effect ofadding some chemical materials on the compressive strength and surfacehardness of dental stone, Eastern Academic Journal, 4,104-110.

[7] Craig RG.(2012) Restorative dental materials. 13th ed, St Louis:Mosby/Elsevier, Pp 185-208.

[8] Heshmati RH, Nagy WW, Wirth CG, Dhuru VB (2002) Delayed linearexpansion of improved dental stone. J Prosthet Dent 88: 26-31.

[9] Lodovici E, Meira JB, Filho LE, Ballester RY (2005) Expansion of high flow mixtures of gypsum-bonded investments in contact with absorbent liners. Dent Mater.; 21: 573-579.

[10] Anusavice KJ (1996) Phillip's science of dental materials. 10th ed, WB Saunders Company, pp: 185-208.

[11] Wise M (2001) Fit of implant supported fixed prostheses fabricated on master casts made from a dental stone and a dental plaster. J Prosthet Dent.; 86(5): 532-538.

[12] Hamdy, T. M., Abdelnabi, A., & Abdelraouf, R. M. (2020). Reinforced dental plaster with low setting expansion and enhanced microhardness. Bulletin of the National Research Centre, 44, 1-7.

[13] Khalaf Hanan Abdul- Rahman, Mohammed Mithaq Radhi (2014) Effect of disinfectant agents on certain physical and mechanical properties of type IV dental stone. J Bagh College Dentistry. 26(1):24-31.

[14] Salem SA, Al-Hadithi KK, Al-Shakhily GA (1997) The effect of disinfectant solutions on some properties of dental stone cast. Iraqi Dent J.; 19: 51-70.