

Influence of Water Type and Water/Powder Ratio on the Strength and Hardness of Type III and IV Gypsum

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ABSTRACT

Casts with a high surface hardness and abrasion resistance are essential for conventional dental prosthesis construction procedures. The water/powder ratio is a critical factor determining the physical and chemical properties of gypsum casts, and any modification to this ratio may alter these properties. This study evaluated the influence of water type and water-powder ratios on the Brinell hardness and the Compressive strength of type III & IV gypsum casts. Three groups of test samples were prepared using type III and type IV gypsum control group - recommended water/powder ratio, and tests groups; 20% and 30% increase in water/powder ratio. The prepared samples were subjected to Compressive strength test and Brinell hardness test using the universal material testing machine. Two-way ANOVA and Tukey's test were used to analyse the data ($\alpha=5\%$). A significant reduction in Compressive strength and Brinell hardness was found in samples prepared with increased water-powder ratios using both the tap water and distilled water. Tap water samples showed significantly lower Compressive strength and Brinell hardness values than the distilled water samples. Chipping was observed in both type III and type IV gypsum samples prepared with an increase in the water powder ratio. Type III gypsum samples exhibited single fracture lines with the recommended water powder ratio whereas type IV gypsum samples had composite fractures. Distilled water samples exhibited higher Compressive strength and Brinell hardness values. Type III and IV gypsum samples prepared with increased water-powder ratio showed statistically significant reduction in the Compressive strength and Brinell hardness values.

Keywords: Dental Gypsum; Compressive Strength; Brinell Hardness; Water Type; Water-Powder Ratio

Abbreviations: MPA: Mega Pascals; psi: Pounds Per Square Inch; ISO: International Organization for Standardization; CHHREC: College Human Health Research Committee; BHN- Brinell Hardness Number

Introduction

Gypsum is one of the materials most frequently used in dentistry to fabricate direct and indirect prostheses [1-5]. Casts with a high surface hardness and abrasion resistance are essential for conventional dental prosthesis construction procedures [1,6]. Type III and IV gypsum are commonly used to fabricate casts for prosthodontics procedures, as they have acceptable mechanical properties such as high resistance, minimal setting expansion, and high surface hardness resistance [1,3,6,7]. In addition, high accuracy is also essential to accurately reproduce details and for dimensional stability [8-12]. The water/powder ratio is a critical factor determining the physical and chemical

properties of a cast, and any modification of this ratio may alter these properties. The possibility of increase in setting time and generating a cast with fewer crystals per volume, lower resistance, and reduced setting expansion is indicated with increase the water/powder ratio [8]. On the other hand, lower water powder ration, reduces the fluidity of the cast, producing inaccurate replication of mould details [8]. As the excess water present within the crystals evaporates, the structure of the set gypsum cast becomes porous, and as the water/powder ratio increases, so does the volume of porosities, reducing the strength of the material after it has dried, because fewer dihydrate crystals are available per unit volume [8,9,13,14]. Any increase in the water-powder ratio consequently increases the amount of water per

unit volume of gypsum, thus decreases the crystallization nuclei per unit volume and therefore increases the setting time of the product [14-17]. Setting expansion is also inversely proportional to water-powder ratio; increase in water powder ratio increases the volume of micro-porosities and consequently increases the setting expansion [18-20].

Type III gypsum should have a minimum compressive strength of 20 Mpa (2900 psi) and type IV gypsum should have a minimum compressive strength of 35 Mpa (5076 psi) one hour after setting, according to the International ISO Standards for gypsum products [21]. These guidelines further recommend use of distilled water to mix gypsum products according to the manufacturer's suggested water/powder ratios [13,21,22]. Various studies have been conducted to assess the effects of different water sources used to mix gypsum products and the results showed that the compressive strength of gypsum decreases as the dissolved ion concentration within the water increases [15,23,24]. The type of water and gypsum products being used determines the water-powder ratio since the wetting ability of the type of water affects the setting time of gypsum [20,25]. Other methods available to improve the strength of gypsum cast include addition of additives and chemicals when mixing the gypsum to alter the setting time and physical properties, resulting in more dense packing of crystals [5,26]. Drying casts in microwave has also been proven to increase the strength of the set cast [3,27]. However, these methods are time consuming and will have additional associated cost.

There is a direct association between the compressive strength of the casts, their surface hardness and resistance to abrasion during handling both in clinic and during the various stages of prosthesis

fabrication at the dental laboratory. The «eye-balling» of the mixture of gypsum when fabricating casts/models is a widespread practice among dental students and dental technicians. Incidences relating to breakage and damage of casts are also frequently encountered by dental students which often requires repeating impression procedures. The recommended water type for mixing gypsum products is distilled water, however dental students and dental technicians use tap water for mixing gypsum as it is readily available. Therefore, this study was conducted to investigate the effects of mixing type III and type IV gypsum using different water types and water/powder ratios on the compressive strength and

Brinell hardness of the set cast.

Methods

Preparation of Samples

One hundred and twenty test samples were prepared using type III gypsum (USG Hydrocal 105, USA) and type IV gypsum (GC FujiRock EP, USA) in total, under ethical approval from College Human Health Research Committee (CHHREC) (ID: 285.20), College of Medicine, Nursing and Health Sciences, Fiji National University, Fiji. The dimensions for compressive test samples were H: 40±0.4mm and D: 20±0.2mm, in accordance with the ISO international guidelines and the dimensions for the Brinell hardness test samples were H: 25mm and D: 60mm as used by a previous study [7] (Figures 1A & 1B). Three groups of test samples were prepared for the type III and type IV gypsum; control group- recommended water/powder ratio (n=40), and tests groups; 20% increase in water/powder ratio (n=40) and 30% increase in water/powder ratio (n=40) (Table 1).

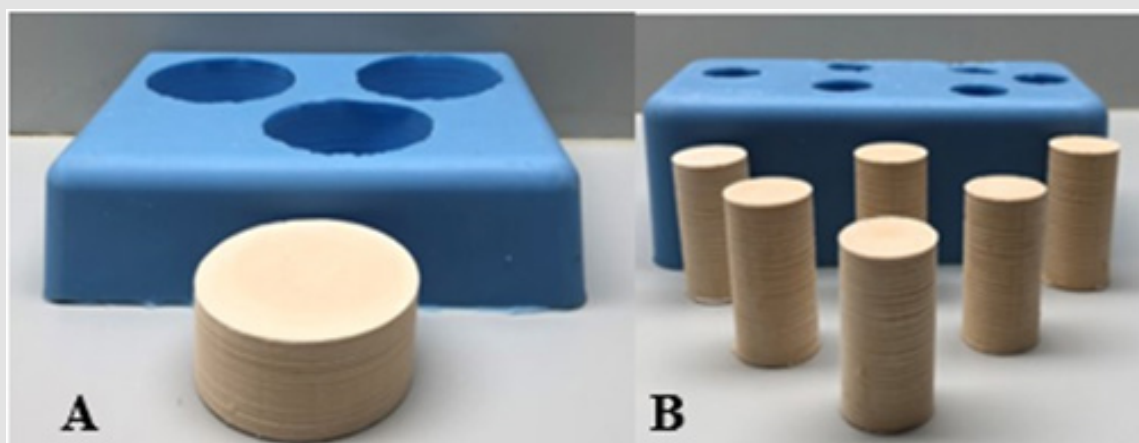


Figure 1: Silicone mold and sample for A- Brinell Hardness test and B- Compressive strength test.

All samples were prepared in a room with temperature maintained at 25 °C ± 2°. Gypsum was weighed on a digital scale (UWE, South Africa), water was measured using a syringe (Changzhou Hansar Medical Technology Co., Ltd. Jiangsu, China) and vacuum mixed

(Renfert Twister Vacuum Mixing Unit, Germany) for 30 seconds. The silicon molds were sprayed with debubbler (Hinrisol, Ernst Hinrich dental GmbH, Goslar, Germany) prior to being filled with the mixed gypsum using a vibrator (Hertz plaster vibrator – Sirio, Meldola,

Italy). A glass slab was placed on the surface of the mold until gypsum completely set to ensure flat and smooth surface. Poured gypsum was allowed to set for one hour before removal from the mold and assessed for elements that would interfere with the Compressive strength test

and Brinell hardness test such as bubbles, cracks and faults. Samples with the above-mentioned imperfections present were discarded. Retrieved samples were allowed to dry at room temperature for 24 hours prior to being subjected to testing.

Table 1: Specimens for Compressive Strength and Brinell hardness tests.

Water Type	Water-Powder Ratio	Water/powder ratio	Compressive Strength		Brinell Hardness	
			Type III	Type IV	Type III	Type IV
Tap water	Recommended	30ml/100g	10	10	10	10
	20% increased	36ml/100g	10	10	10	10
	30% increased	39ml/100g	10	10	10	10
Distilled water	Recommended	20ml/100g	10	10	10	10
	20% increased	24ml/100g	10	10	10	10
	30% increased	26ml/100g	10	10	10	10

Measuring Compressive Strength

The samples were subjected to Compressive strength test on a WP-300 universal material testing machine (GUNT, Hamburg, Germany) with a load cell of 2,000 kgf/cm² and a displacement velocity of 0.5 mm/min. The load on the samples were slowly and steadily increased, until the fracture lines were observed. The Compressive strength test data was generated by the data acquisition system of the WP-300 universal material testing machine (GUNT, Hamburg, Germany).

Measuring Brinell hardness

The samples were subjected to Brinell hardness test on a WP-300 Universal Material Testing Machine (GUNT, Hamburg, Germany) with a 10mm ball indenter with a force of 2500N for 30 seconds. Diameter of the indentation produced on the type III and type IV samples were measured by using a WP-300.12 measuring magnifier (GUNT, Hamburg, Germany). The average values of the diameter of the indentation of the 10 samples of each group was used to calculate the Brinell Hardness Number (BHN) using the following formula:

$$\text{Brinell hardness} = (2 \times 0.102 \times F) / \pi D(D - \sqrt{D^2 - d^2})$$

Where:

F = Force

D = Diameter of Indenter

d = Diameter of indentation

The data sets were then analysed using the statistical software (IBM SPSS Version 26, USA). Descriptive statistics with mean, standard deviation for all tests and groups were computed. The statistical differences between the tested materials were assessed with Two-way ANOVA and Tukey's tests ($\alpha=5\%$).

Results

Compressive Strength

The highest mean Compressive strength (118.24 ± 16.47MPa) for type III gypsum was recorded in the samples prepared with

distilled water using the recommended water powder ratio and the lowest compressive strength (70.81 ± 10.08MPa) was recorded in the samples prepared with tap water with 30% increase in water-powder ratio (Table 2). A statistically significant reduction in mean Compressive strength was recorded in tap water samples prepared with 30% increased water-powder ratio (p= 0.001) and for both 20% (p= 0.023) and 30% (p= 0.000) increased water-powder ratio samples prepared with distilled water for the type III gypsum samples (Table 3). The highest Compressive strength (181.20 ± 52.13MPa) for the type IV gypsum was recorded in the samples prepared using the recommended water/powder ratio with distilled water and the lowest compressive strength (130.15 ± 13.98MPa) was recorded in the samples prepared using tap water with 30% increased water-powder ratio. A significantly lower mean Compressive strength in type IV gypsum was recorded in samples prepared using 30% increased water-powder ratio (p= 0.038) with tap water and in samples prepared with 30% increase in water-powder ratio prepared using distilled water (p= 0.006) (Table 3).

Brinell Hardness (BHN)

For type III gypsum, highest Brinell hardness mean value (12.13 ± 0.52 BHN) was recorded in the samples prepared using distilled water with the recommended water powder ratio and the lowest mean Brinell hardness value (7.41 ± 0.35 BHN) was recorded in the samples prepared with tap water using 30% increase in water-powder ratio (Table 4). For type IV gypsum, the highest mean Brinell hardness value (31 ± 2 BHN) was recorded in the samples prepared using distilled water with the recommended water powder ratio and the lowest mean Brinell Hardness value (18 ± 1 BHN) was recorded in the samples prepared with tap water with 30% increase in water-powder ratio (Table 4). A significantly lower mean Brinell hardness value was recorded for both the 20% increase water-powder ratio (p= 0.000) and 30% increase water-powder ratio (p= 0.000) samples prepared using tap water. Similarly, the mean Brinell hardness value was significantly lower for both the 20% increase water-powder ratio (p= 0.023) and 30% increase water-powder ratio (p= 0.000) samples that were prepared with distilled water (Table 5).

Table 2: Mean Compressive Strength for type III and IV gypsum.

	Water Type	Water-Powder Ratio	Mean Compressive Strength	
			(MPa)	Std. (±)
Type III	Tap water	Recommended	104.06	19.76
		20% Increase	86.03	20.3
		30% Increase	70.81	10.08
	Distilled water	Recommended	118.24	16.47
		20% Increase	92.44	16.93
		30% Increase	80.86	17.66
Type IV	Tap water	Recommended	171.56	20.01
		20% Increase	147.45	34.73
		30% Increase	130.15	13.98
	Distilled water	Recommended	181.2	52.13
		20% Increase	156.8	26.55
		30% Increase	130.66	15.55

Note: Type III- The error term is Mean Square (Error) = 297.93; Type IV -The error term is Mean Square (Error) = 911.15.

Table 3: Comparison of Compressive strength values of Type III & IV gypsum.

Water Type	Water Powder ratio	Mean (MPa)	Std. (±)	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Type III gypsum						
Recommended distilled water	Recommended tap water	14.18	7.72	0.451	-8.62	36.99
Recommended tap water	20% Increase tap water	18.03	7.72	0.198	-4.77	40.84
	30% Increase tap water	33.25*	7.72	0.001*	10.44	56.06
Recommended distilled water	20% Increase distilled water	25.79*	7.93	0.023*	2.36	49.22
	30% Increase distilled water	36.34*	7.54	0.000*	14.06	58.62
Type IV gypsum						
Recommended distilled water	Recommended tap water	9.64	13.5	0.979	-30.24	49.52
Recommended tap water	20% Increase tap water	24.11	13.5	0.483	-15.78	64
	30% Increase tap water	41.41*	13.5	0.038*	1.53	81.3
Recommended distilled water	20% Increase distilled water	24.4	13.5	0.47	-15.49	64.28
	30% Increase distilled water	50.54*	13.5	0.006*	10.66	90.42

Note: Type III- The error term is Mean Square (Error) = 297.93; Type IV -The error term is Mean Square (Error) = 911.15. * Significance level (p ≤ 0.05).

Table 4: Mean Brinell hardness for Type III and IV gypsum.

Material	Water Type	Water-Powder Ratio	Mean Brinell Hardness	
			(BHN)	Std. (±)
Type III	Tap Water	Recommended	11.4	0.52
		20% Increase	7.77	0.35
		30% Increase	6.65	0.27
	Distilled Water	Recommended	12.13	0.5
		20% Increase	8	0
		30% Increase	7.41	0.71
Type IV	Tap Water	Recommended	28	1
		20% Increase	20	1
		30% Increase	16	1
	Distilled Water	Recommended	31	2
		20% Increase	20	1
		30% Increase	18	1

Note: Type III- The error term is Mean Square (Error) = 0.24; Type IV -The error term is Mean Square (Error) = 1.39.

Table 5: Comparison of mean Brinell Hardness of Type III & IV Gypsum with different Water Type and Water Powder Ratio.

Water Type	Water Powder Ratio	Mean Diff (BHN)	Std. (±)	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
Type III gypsum						
Recommended distilled water	Recommended tap water	0.73*	0.22	0.02*	0.09	1.38
Recommended tap water	20% Increase tap water	3.40*	0.22	0.00*	2.76	4.04
	30% Increase tap water	3.99*	0.22	0.00*	3.34	4.63
Recommended distilled water	20% Increase distilled water	4.40*	0.22	0.00*	3.74	5.06
	30% Increase distilled water	5.35*	0.21	0.00*	4.72	5.98
Type IV gypsum						
Recommended distilled water	Recommended tap water	2.20*	0.53	0.001*	0.64	3.76
Recommended tap water	20% Increase tap water	8.40*	0.53	0.00*	6.84	9.96
	30% Increase tap water	10.60*	0.53	0.00*	9.04	12.16
Recommended distilled water	20% Increase distilled water	10.50*	0.53	0.00*	8.94	12.06
	30% Increase distilled water	14.70*	0.53	0.00*	13.14	16.26

Note: Type III- The error term is Mean Square (Error) = 0.24; Type IV -The error term is Mean Square (Error) = 1.39. * Significance level ($p \leq 0.05$).

Fracture Lines

For type III gypsum, the recommended water-powder ratio samples presented a single medial longitudinal fracture line with mild chipping for both tap water and distilled water samples after being subjected to compressive strength testing. However, as the water-powder ratio increased, chipping of the samples increased,

and the fracture lines were no longer centered in the medial aspect (Figure 2). For type IV gypsum, the recommended water-powder ratio samples presented composite fractures. These composite fractures had longitudinal, and oblique fractures causing the samples to break into multiple fragments (Figure 3). Interestingly, the 20% and 30% increase in the water-powder ratio type IV gypsum samples exhibited single fracture lines with chipping (Figure 3).

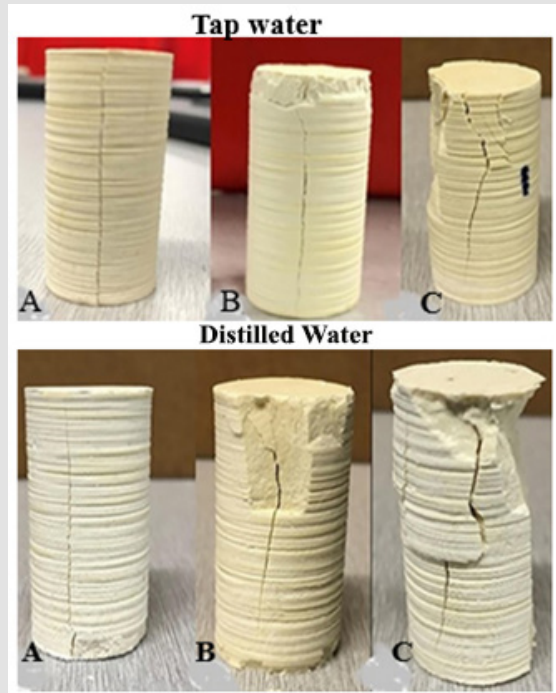


Figure 2: Type III gypsum fracture lines.

- A. Recommended water powder ratio,
- B. 20% increased water powder ratio and
- C. 30% increased water powder ratio.

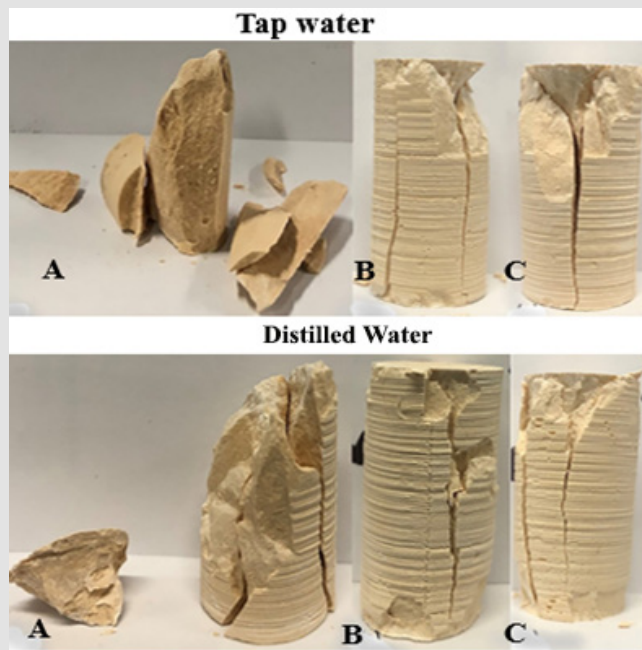


Figure 3: Type IV gypsum fracture lines;

- A. Recommended water powder ratio,
- B. 20% increased water powder ratio and
- C. 30% increased water powder ratio.

Discussion

This study investigated the effect of different water-powder ratios and water types on the Compressive strength and Brinell hardness on type III and type IV gypsum. The study used one commercial brand of type III and type IV gypsum with two different types of water and three different water-powder ratios. The results showed that water-powder ratio and the type of water used to mix gypsum can potentially alter the mechanical properties of both type III and type IV gypsum, specifically: Compressive strength and Brinell hardness. Both, type III and type IV gypsum samples prepared using distilled water following manufacturer's recommended water-powder ratio had higher Compressive strength values. This result is similar to the findings of two earlier studies by Jayaprakash, et al. [15] and Musa, Nafea, & Hasan [28] which also recorded a significantly lower Compressive strength and Brinell hardness in tap water samples [15,28]. The presence of ion concentration in tap water could be a possible reason for the reduced Compressive strength in tap water samples. Ions contained within the mixing solution alters the gypsum crystalline structure that affect the gypsum crystal's ability to intermesh by reducing the formation of dihydrate crystals as less water molecules will react with hemihydrate, resulting in a weaker substructure [15,24]. Ion content in mixing solutions can also act as retarders and accelerants which alters the setting time and strength of the gypsum [28,29]. Faster setting times have been associated with increased ion concentrations in water samples, resulting in less time available for the formation dihydrate crystals; causing an irregular crystal formation resulting in reduced inter crystalline cohesion [15,29]. However, due to absence of ion content in distilled water, the dihydrate crystal formation is regular, relatively non-porous and denser [29]. The mean Brinell hardness value and Compressive strength recorded for both type III and IV gypsum samples prepared using tap water for the current study is consistent with an earlier study by Sabooni and Ghanbarzadeh [23] which also attributed the decrease in the strength and surface hardness of gypsum to the presence of salts or ions in the water [23]. On the contrary, Puspitasari, et al. [24] findings showed that 'very hard water' samples, had higher Compressive strength values than that of the 'hard water' sample, [24]. The investigators stated this could be due to the variation in the concentration of ions and salts in the water type used.

The overall mean Compressive strength and Brinell hardness values for samples tested in the current study was higher than a few earlier studies by Tavarez et al., (2014), da Silva et al. (2012), and Jayaprakash, et al. [8,10,15]. The variation in the time of testing after samples were prepared could be the possible reason for the lower strength and hardness values for the study by Tavarez et al., (2014) as the testing was conducted one hour after the samples were prepared [8]. Although the other two studies prepared the samples following the same method to the current study and tested them 24 hours later, they utilized different brands of gypsum products, which could have had an adverse effect on the strength and hardness values due to difference in the chemical compositions. Test samples prepared by da Silva et al. (2012) study used Sherapremium (SHERA Werkstoff-

Technologie GmbH & Co. KG Espohlstraße, Lemförde, Germany) and Duron Iv (Dentsply, Pennsylvania, USA) [10]. According to the material safety data sheet for Sherapremium [30], the material contains 80-100% calcium sulfate whereas GC Fujirock EP (GC America, Illinois, USA) used in the current study contained 90-100% calcium sulfate [31]. The study by Jayaprakash, et al. [15] used Kalrock (Kalabhai, Mumbai, India) and Pearl Stone (Asian Chemicals, Rajkot, India), however calcium sulfate composition could not be determined as the material safety data sheet for the brand was not available online [15]. Therefore, it can be assumed that reduced gypsum dihydrate content lowers the material's density, thereby reducing the by product's Brinell hardness and Compressive strength [18]. The sample preparation technique for the current study was standardized to allow minimal room for errors; gypsum was mixed using a fully automated vacuum mixer for thirty seconds and all silicone moulds were sprayed with debubbler prior to being filling with gypsum mixture to prevent the formation of air bubbles in the samples. Earlier studies by Jørgensen & Kono (1971) [18] and Gandra & Aveiro (2011) [19] showed that presence of air porosities reduces the density of the gypsum material therefore lowering the Compressive strength [18,19]. However, none of the previous studies have stated use of any wetting agents on the silicone molds. This can be tested and verified through future studies by comparing samples with and without a wetting agent being sprayed on the silicon molds before filling the gypsum mixture.

A similar trend was recorded for both type III and type IV gypsum; as the water powder ratio was increased to 20% and 30% an inversely proportional relationship was observed in both the Compressive strength and Brinell hardness values. This might be explained by the fact that with a higher water-powder ratio, the destruction of bonds between small and bigger crystals through precipitation occurs, resulting in micro-porosities that reduces the material's ability to resist stress [8]. An increase in water powder ratio increases the setting time, resulting in weaker gypsum product due to an increase in porosity since fewer dihydrate crystals are available per unit volume and when the excess water evaporates during drying, the space occupied previously by water become porosities [16,18,20]. Generally, more chipping was observed in the tap water samples as the water powder ratio increased than the distilled water samples with type III gypsum. Composite fractures were seen in both the distilled water and tap water type IV gypsum samples with recommended water powder ratio using. According to an earlier study by Tavarez et al., (2014) , as the hardness of the material increases, it becomes more friable and hence, as the water powder ratios increased, the composite fractures in the type IV gypsum samples became single fracture lines and chipping was observed as the material was failing at a lower force as compared to that of the recommended water-powder ratio samples [8].

The current study is used a single brand of type III and type IV gypsum products and hence comparisons across different brands could not be made. Human error or inter-examiner variability is another limitation that needs to be mentioned since the universal material testing machine did not have automatic loading and force

was supplied manually by different examiners which may have resulted in difference in the pacing of the force being loaded. Despite the availability of digital technology for fabricating dental prostheses, conventional methods will likely continue to be used widely, particularly in underdeveloped nations, demanding the manufacture of high-quality dental casts. Water-powder ratios and the type of water used in preparing gypsum casts can either enhance the physical properties of the material or degrade them. Despite being readily available, tap water does not provide the best practical outcomes when used to mix gypsum products in terms of strength and hardness of dental casts. Therefore, the recommended water type and water powder ration should be used as a standard practice. Alteration of basic manufacturer's instructions can result in obtaining a cast with unfavorable strength and quality.

Conclusion

Within the limitations of this study,

1. Type III and IV gypsum samples prepared with distilled water showed higher Compressive strength and Brinell hardness values.
2. Type III and IV gypsum samples prepared with 20% and 30% increase in water-powder ratio showed statistically significant reduction in the Compressive strength and Brinell hardness values.

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Conflict of Interest

We have no conflicts of interest to disclose.

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