

## EVALUATION OF THE DEGRADATION OF DENTAL RESTORATIVE MATERIALS CENTION-N, EQUIA, TETRIC N CERAM IN ARTIFICIAL SALIVA AT DIFFERENT TIME INTERVALS USING FTIR (FOURIER TRANSFORM INFRARED SPECTROSCOPY) – AN IN VITRO STUDY.

### Dental Science

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### ABSTRACT

**Aim-** To evaluate and compare the degradation of 3 restorative material Cention-N, Equia, Tetric N Ceram in artificial saliva at 4 different time interval using FTIR (Fourier Transform Infrared Spectroscopy). **Methodology-** 32 Teflon molds of standard size 2 x 2 x 2 mm were made to create blocks of the material of the same size. 32 blocks of each material were made (n=32). Group 1 = Cention -N, Group 2 = Equia, Group 3 = Tetric N Ceram. It was then placed into small polypropylene vials containing 2 ml of artificial saliva at 24 hrs, 1 week, 1 month, 2 month (n=8). At each time interval the material block were pulverized and molded into a pellet form using a pressurized apparatus and subjected to the FTIR spectroscopy. The graphical output was quantified and compared between each material at different time interval. **Results-** Group 1 (Cention N) and Group 2 (Equia) showed degradation in all the time periods. Group C (Tetric N Ceram) also showed degradation in all the time periods but there was no statistically significant increase in degradation between the time periods

### KEYWORDS

Cention -N, Equia Forte, Tetric N Ceram

### INTRODUCTION

The development of hydrolytically stable dental material is an important goal in dental material research. Restorations may either be exposed continuously to chemical agents such as those found in saliva or food. The diffusion of moisture through the resin based materials may also lead to nucleation and growth of micro cracks at the interface and in the resin.

Recently a new restorative concept has been marketed (Equia, G. CTokio Japan). This system consisting of highly viscous conventional GIC combined with a novel nanofilled coating material (Equia formerly known as G-Coat Plus). Compounded nano fillers are thereby intended to protect the system against adhesive wear.<sup>[1]</sup>

Tetric N-Ceram Bulk Fill (Ivoclar Vivadent) was recently introduced with the claim that it would substitute both conventional nonflowable composite and bulk-fill flowable composite that needed an increment of 2 mm when using the incremental layering technique. As per the manufacturer's commercials, this new composite will achieve full-depth bulk fill up to 4 mm without a superficial capping layer. The Tetric N-Ceram Bulk Fill contains a shrinkage stress reliever to minimize polymerization shrinkage. It comprises of an overall standard filler content of approximately 75% by weight, 61% volume and 17% polymer fillers or biofiller.<sup>[2]</sup>

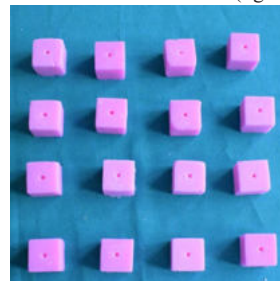
Cention N (Ivoclar Vivadent) is a recently introduced tooth coloured basic filling material for bulk placement in retentive preparation with or without the application of an adhesive. It is an "alkasite" restorative which is a new category of filling material, like compomer or ormocer and is essentially a subgroup of the composite resin. Cention -N is a UDMA based self curing powder liquid restorative with optional additional light curing. The liquid comprises of dimethacrylate and the powder contains various glass fillers, initiator and pigments. It contains alkaline glass filler capable of releasing fluoride, calcium and hydroxide ion.

Changes in the chemical structure and the disintegration of the bonds of dental materials can be studied by FTIR after storage in salivary medium and is one of the acceptable methods to understand the degradation potential of the dental material.

The availability of research evaluating and comparing the degradation of Cention N, Equia, Tetric N Ceram in artificial saliva is very limited. Hence aim of the study is to evaluate and compare the degradation of 3 recently introduced restorative materials Cention-N, Equia, Tetric N Ceram using FTIR

### MATERIALS AND METHODS

Teflon molds of standard size 2 x 2 x 2 mm of 96 blocks was made. With this Teflon models blocks of test materials (Cention-N, Equia forte, Tetric N Ceram) of the same size was created. (figure 1)



The block of material was made by manipulating the restorative test materials as per manufacturer's instructions. figure 1

Cention-N was mixed on a paper pad using an agate spatula and the mixed material was condensed into the molds. Equia forte which comes as a capsule was mixed in an amalgamator and dispensed in to the Teflon mold with the help of loading gun. Tetric N Ceram nanohybrid composite was condensed in to the Teflon mold and was light cured for 20 seconds using light cure unit. After setting, the blocks of the material were removed and placed into small polypropylene vials containing 2 ml of artificial saliva. A total of 32 blocks of each material were made (n=32).

### The test materials were classified in to 3 groups:

- Group 1 = Cention -N
- Group 2 = Equia forte
- Group 3 = Tetric N Ceram

The samples were subdivided into 4 subgroups of 8 each (n=8) to evaluate and compare the degradation of 3 restorative materials in 4 different time interval of 24 hours, 1 week, 1 month, 2 months.

Subgroup A	-	sample subjected to 24 hours of placement in artificial saliva
Subgroup B	-	sample subjected to 1 week of placement in artificial saliva
Subgroup C	-	sample subjected to 1 month of placement in artificial saliva
Subgroup D	-	sample subjected to 2 month of placement in artificial saliva

## ANALYSIS

At each time interval the material blocks were pulverized and molded into a pellet form using a pressurized apparatus before subjecting to the FTIR spectroscopy. The spectroscopy chart would characterize the bond and the ions of the material and the material degradation would be observable as the changes in the spectroscopy. The graphical output was quantified and compared between each material at different time intervals.



figure 2 (FTIR spectroscopy)

## RESULTS AND STATISTICAL ANALYSIS

**Table 1- Descriptive Statistics with absorbance against wavelength (900-1100)**

Groups	900-1100 wave number	N	Minimum	Maximum	Mean	Std. Deviation
Cention	24 Hours	8	72.73	146.91	126.1288	25.21646
	1 Weeks	8	60.45	109.32	94.0675	20.92109
	1 Months	8	13.09	178.89	74.9475	77.41620
	2 Months	8	25.56	102.09	57.2775	32.07671
Equia	24 Hours	8	115.15	171.60	161.4650	19.30653
	1 Weeks	8	130.95	163.00	149.3750	13.72931
	1 Months	8	88.92	124.84	102.4738	13.75485
	2 Months	8	60.45	109.32	92.8175	20.16465
Composite	24 Hours	8	13.54	194.07	140.7063	54.16390
	1 Weeks	8	18.10	176.52	136.2700	48.80531
	1 Months	8	95.26	145.48	124.3963	16.69326
	2 Months	8	102.40	129.40	113.0850	8.42956

Group A (Cention N) showed degradation in all the time periods (1day,1 week,1month,2 month). At wave numbers from 900-1100 Group A (Cention N) has got the mean value of 126.1288, 94.0675, 74.9475, 57.2775 at 1 day,1week, 1month, and 2 month. The highest mean value was found at 1 day (126.1288) and lowest mean value at 2 months (57.2775).

There is significant difference between the time periods for Cention N.

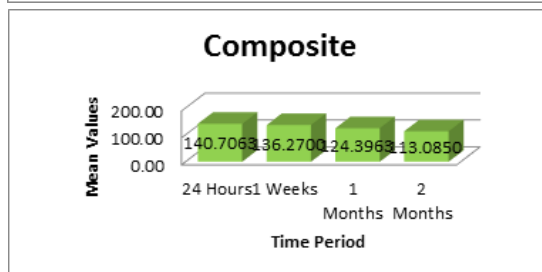
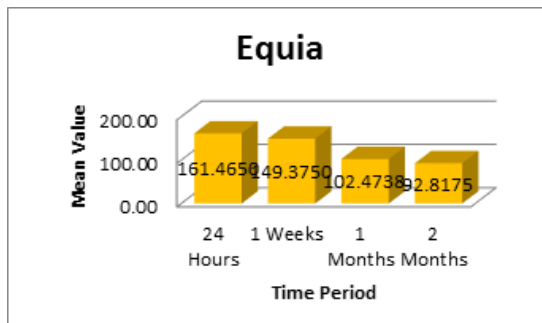
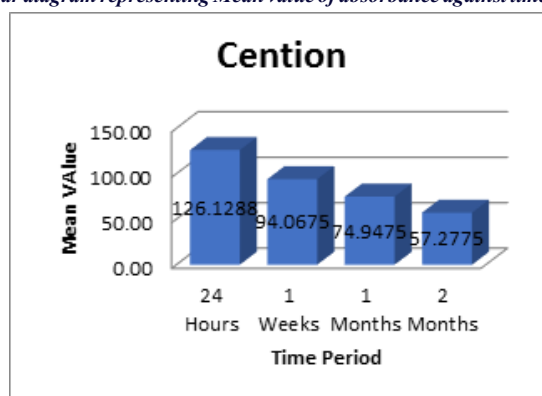
Group B (Equia Forte) showed degradation in all the time periods (1day, 1 week, 1month,2 month) At wave numbers from 900-1100 Group B (Equia Forte) has got the mean value of 161.4650, 149.375, 102.473, 92.817 at 1 day,1week, 1month, and 2 month. The highest mean value was found at 1 day (161.465) and lowest mean value at 2 months (92.817). There is significant difference between the time periods for Equia Forte.

Group C (Tetric N Ceram) also showed degradation in all the time periods (1day,1 week,1month,2 month) but there was no significant increase in degradation between the time periods. At wave numbers from 900-1100 Group C (Tetric N Ceram) has got the mean value of 140.706, 136.270, 124.396, 113.085 at 1 day,1 week, 1month, and 2 month. The highest mean value was found at 1 day (140.706) and lowest mean value at 2 months (113.085).

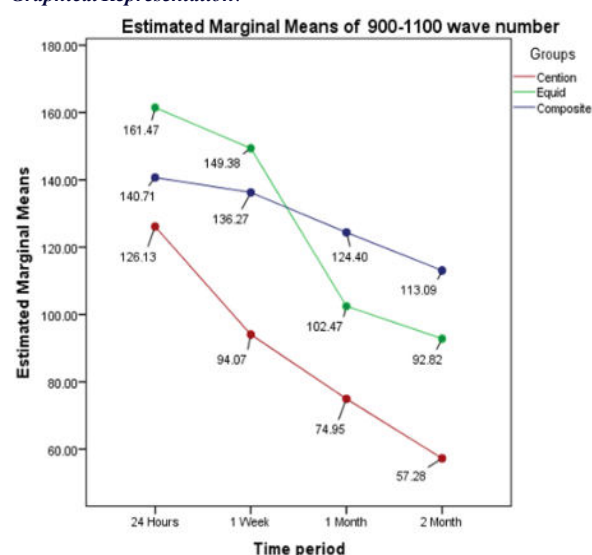
In 1 day there was no significant difference in degradation between the three groups.

In 1 week,1 month ,2month there was statistically significant difference between Group A(Cention N) and Group B(Equia Forte) & Group A (Cention N) & C (Tetric N Ceram) but no statistically significant difference was there in between group B(Equia Forte) and Group C (Tetric N Ceram).

## Bar diagram representing Mean value of absorbance against time



## Graphical Representation:



From the above line plot we can see that Equia has the high values in 900-1100 wave number for at the time point 24 hours. At 2 month Cention has low value in 900-1100 wave number than Equia and Tetric N ceram.

ANOVA analysis was done to find out if there is any significant difference in 900-1100 wave number between the time periods for each group. Post hoc analysis and Tukey HSD was also done since there was a significant difference between the time periods.

ANOVA analysis for group 1 (cention) showed p value  $0.030 < 0.05$ , group 2 (equia) showed p value  $0.000 < 0.05$  and for group 3 (Tetric N ceram) was  $0.467 > 0.05$ .

## DISCUSSION

Long-term performance of dental restorative materials is determined by their physical and mechanical properties. In attempting to understand the phenomena involved in the biodegradation of restorative materials in vitro models may be most important in providing an insight into the fundamental mechanism of biodegradation.<sup>[3]</sup>

Many of the restorative materials that were tested in earlier studies have been replaced with improved formulations. In this context, although clinical trials are the ideal method of predicting restoration behaviour, laboratory studies may be useful in providing information on the fundamental mechanisms of degradation.<sup>[4]</sup>

Cention N is an "alkasite" restorative. Alkasite refers to a new category of filling material, which like compomer or ormocer materials is essentially a subgroup of the composite material class. This new category utilizes an alkaline filler, capable of releasing acid-neutralizing ions. It is self-curing with optional additional light-curing. It is radiopaque, and releases fluoride, calcium and hydroxide ions. The liquid comprises dimethacrylates and initiators, whilst the powder contains various glass fillers, initiators and pigments.

EQUIA (GC) is a new glass ionomer restorative system. The manufacturers claim that the material has increased fracture toughness, flexural strength, and flexural fatigue resistance. The coating agent added to the surface of the glass-ionomer of the EQUIA system contains a nanofilled resin that contributes significantly to the increased resistance of the material to mechanical forces. Adhesion of EQUIA to dentin occurs by a micromechanical interlocking and a chemical bonding with the hydroxyapatite in enamel and dentin. The more voluminous glass fillers of EQUIA Forte were supplemented by smaller, highly reactive fillers that strengthen the restoration.

Tetric N Ceram Bulk Fill is a nanohybrid composite with a monomer matrix containing dimethacrylates (20-21% weight). The fillers contain barium glass, ytterbium trifluoride, mixed oxide, and prepolymer (78%–81% by weight). The total content of inorganic fillers is 76-77% weight. Tetric N Ceram Bulk Fill contains in its composition an inhibitor of sensitivity to light and thus provides prolonged time for modeling of filling, an inhibitor of shrinkage stress in order to achieve optimal marginal seal, and Ivocerin, polymerization photoinitiator allowing curing of 4 mm layers of material.

FTIR spectroscopy is a widely used technique for investigating materials in the gaseous, liquid or solid phase. It is based on the interaction between electromagnetic radiation and natural vibrations of the chemical bonds among atoms that compose the matter.

In our study we had used FTIR to study the chemical composition and reaction mechanism occurring in Tetric N Ceram composite, cention and Equia. FTIR is a valuable tool in detecting the disintegration of different bonds. The machine can emit a wide range of infrared radiation and it works by finding absorption of energy in a wavelength or wave number to investigate the chemical structure of the material being tested.

Raman and IR spectroscopy is the study of the scattering, reflection, absorption or transmission of radiation. However, some consider that both methods have been evaluated to be complementary in molecular vibrational analysis.<sup>[6]</sup> Preliminary Raman studies with Fuji IX have also shown that without submersion in water only about 50% of the acid can react. With water immersion this increases to more than 75% in under 24 h. The FTIR spectrum at 24 h in therefore corresponds with approximately 75% acid neutralisation.

Our study for the FTIR examinations showed that all of the materials examined, with the exception of Tetric N Ceram showed spectral changes and were not chemically stable. All of the materials after immersion showed chemical changes in the whole spectra with an increase in transmission. The tetric n ceram composite appeared to be most chemically stable of the materials examined. This result was in accordance with H.K YIP et al<sup>[5]</sup>.

The FTIR results for all of the specimens showed chemical changes with an increased transmittance at different wave numbers as the immersion times increased. We used artificial saliva as the oral fluid to look at Cention- N, Equia and Tetric N Ceram nano hybrid composite. At wave numbers from about 900 to 1100  $\text{cm}^{-1}$  the cention -n and equia forte graph showed a considerable drop in transmittance. The drop became less significant as the immersion time increased<sup>1</sup> which indicated that the amount of the bonding inside the material decreased after the specimen was immersed in artificial saliva for longer periods.

When quickly viewing the spectra of the resin composites, the spectra look almost identical. They have the same general band positions, and the differences between them seem minute or even null. A previous Fourier transform infrared (FTIR) spectroscopy study demonstrated that the visible light-curing process, greatly reduces the salt formation (acid-base reaction) rate during the early setting stages, which is likely due to rapid polymer network formation. In our study the Tetric N Ceram showed the least salivary disintegration the combination of a nanohybrid with the prepolymer technology, used in Tetric-N Ceram. According to the manufacturer Tetric N Ceram mainly consists of prepolymerized and milled microfillers, ytterbium fluoride particles and nanofillers.<sup>[1]</sup> Nanohybrid composites (Tetric N ceram) have high filler content and monomers which imparts the mechanical properties of bond strength, less polymerization shrinkage, polishing properties.

## CONCLUSION

Within the limitation of this in vitro study, FTIR examinations showed that the materials examined showed spectral changes and were not stable. All the materials showed chemical changes in the spectra. The resin composite appeared to be the most stable.

Cention N showed degradation in all the time periods with significant increase in degradation over time. Equia Forte showed degradation in all the time periods with significant increase in degradation over time. Tetric N Ceram also showed degradation in all the time periods but there was no significant increase in degradation between the time periods. In 1 day there was no significant difference in degradation between Cention N, Equia Forte and Tetric Ceram. In 1 week, 1 month, 2 month there was statistically significant difference in degradation between all the materials tested except Equia Forte and Tetric N Ceram.

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