INTRODUCTION
Dental caries is still considered to be one of the most prevalent chronic diseases affecting the human race. The progression or reversal of the dental caries and secondary caries process depends on the balance between pathological and protective factors. Fluoride has been identified as one of the most protective remineralizing factors, which tilts the caries balance towards the positive side 1.

Fluoride is well documented as an anticariogenic agent. A variety of mechanisms are involved in the anticariogenic effects of fluoride, including the reduction of demineralization, the enhancement of remineralization, the interference of pellicle and plaque formation and the inhibition of microbial growth and metabolism 2.

Fluoride can be made available to tooth surfaces through several methods including dentifrices, mouth rinses, fluoridated water intake, gel and foam. Additionally, fluoride can be available to a tooth surface via fluoride release from restorative materials in close proximity. Today, there are several fluoride containing dental restoratives available in the market including conventional glass ionomer cements (Fuji IX GP, GC Corp.; Ketac N100, 3M ESPE ) and a composite resin (Gradia Direct X, GC Corp) before and after exposure to mouthwash and toothpaste in which a fluoride ion-selective electrode was used to measure fluoride concentrations.

Materials and Methods: Ninety standardized discs of the studied materials were fabricated with dimension of (8 mm diameter and 2 mm thickness) from cylindrical brass moulds suspended in 8ml polyethylene vial. Fluoride released from the materials was measured at 1st, 2nd, 7th 14th and 30th days. At 30th day the specimens were divided into three groups. Specimens in the control group were stored in deionized water. For the other two specimens were exposed fluoridated toothpaste and fluoridated mouthwash. After refluoridation, fluoride amount was measured at 32nd, 39th, 46th, and 53th days.

Results: The release of fluoride was highest on day 1 (90.097) for Fuji IX and (64.819) for Ketac N then there was a sudden fall on day 2 and gradual constant decrease over time, the lowest value is in the thirty days which is (18.86) for Fuji IX, (10.711) for Ketac . While Gradia X shows lower release and leveling off thereafter without any sudden release or decrease in which the fluoride ion released at constant low level. After treatment solutions, there is no effect of fluoride recharge ability by de ionized water but there is a significant increase in fluoride release after treatment with toothpaste and mouthwash, in 32th day which is (36.654) for Fuji IX, (26.768) for Ketac N and (4.160) for Gradia X but the mean value dropped to a constant, low level at 39th and 46th days.at day 53th when refloefuridation is administered on daily basis the rate of fluoride ion release increased significantly.

Conclusions: The three fluoride-containing dental materials used were able to release and exhibited different fluoride release patterns depending on their compositions. After exposure to fluoridated mouthwash and toothpaste, all materials were recharged and continued releasing fluoride, in which recharge ability from the toothpaste group was found to be more successful than the mouthwash group.

So the objectives of this study were to evaluate and compare the concentration of fluoride release from three fluorides releasing esthetic restorative materials and assessing the recharge-ability after exposing to topical fluoride containing agents.

MATERIALS AND METHODS
Sample Preparation:
Ninety standardized discs of the studied materials were fabricated with dimension of (8 mm diameter and 2 mm thickness) from cylindrical brass moulds. All disc samples were made in such a way to be of approximately equal thickness and size using digital venire caliper2.

A mylar matrix strip was first secured on a glass slide to provide the base for the mould. The restorative material was mixed according to the manufacturer's instruction in which for Fuji IX GP was activated just before mixing, set into the amalgamator and mixed for 10 seconds at high speed. For Ketac N the paste was dispensed onto the mixing pad and mixed together for 20 seconds using the plastic cement spatula until the uniform color was achieved and for Gradia Direct X the necessary amount of the composite material was dispensed from the syringe onto the mixing pad, plastic spatula was used to place it into the mold4.

After placement the surface of the restorative material was covered with a second mylar strip and another glass slide was placed over the strip under a weight of 200g for 1 minute until the slide touch the mould completely to extrude excess material and to ensure void free sample discs. All samples of Keatc nano GIC and Gradia X composite were polymerized by an LED Device for 20 seconds from top and 20 seconds from bottom, respectively. The distance between the light source and the specimen was standardized by the use of a 1 mm glass slide in which the end of the curing light was in contact with the cover glass.

ABSTRACT
Aim: The aim of this study was to investigate the fluoride release and fluoride recharge behaviors of two glass ionomer cements ( Fuji IX GP, GC Corp.; Ketac N100, 3M ESPE ) and a composite resin (Gradia Direct X, GC Corp) before and after exposure to mouthwash and toothpaste in which a fluoride ion-selective electrode was used to measure fluoride concentrations. Materials and Methods: Ninety standardized discs of the studied materials were fabricated with dimension of (8 mm diameter and 2 mm thickness) from cylindrical brass moulds suspended in 8ml polyethylene vial. Fluoride released from the materials was measured at 1st, 2nd, 7th 14th and 30th days. At 30th day the specimens were divided into three groups. Specimens in the control group were stored in deionized water. For the other two groups, the specimens were exposed fluoridated toothpaste and fluoridated mouthwash. After refloefuridation, fluoride amount was measured at 32th, 39th, 46th, and 53th days.

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Conclusions: The three fluoride-containing dental materials used were able to release and exhibited different fluoride release patterns depending on their compositions. After exposure to fluoridated mouthwash and toothpaste, all materials were recharged and continued releasing fluoride, in which recharge ability from the toothpaste group was found to be more successful than the mouthwash group.

KEYWORDS: Fluoride release, Recharge, Glass ionomer, composite.
slide during the light polymerization process 7,8. While for Fuji IX GP conventional GIC was allowed to set inside the mould between the glass slides covered by the mylar strip for ten minutes and then after complete setting the set discs were gently pushed out of the mould 7. The steps of the methodology can be summarized by the following figures:

Fluoride concentration measurement using ion-selective Electrode:

Each sample was suspended in 8 ml deionized water in a polyethylene test tube. During the entire experiment, all samples were incubated at a constant temperature of 37±0.5°C and relative humidity of 100%.

Fluoride measurement was done using a combination of fluoride ion electrode coupled to ion analyzer. In the first part of the study the quantities of released fluorides were measured in the following time periods: 1st, 2nd, 7th, 14th and 30th days.

In the second part of the study, the 30 disc samples of each material will be divided into three group's ten of each

Group 1: No treatment as control.
Group 2: Treatment with fluoridated mouthrinse (Sensodyne Pronamel mouthwash)
Group 3: Treatment with fluoridated toothpaste (fluor kin toothpaste)

For the control group, the samples were stored in deionized water. For the other two groups, the samples were exposed to fluoridated toothpaste and mouthwash for three weeks. Refluoridation was done once a week during the first two weeks and on a daily basis during the third week for 4 minutes which is equivalent to two times of 2 minutes daily use. Fluoride measurement of the recharged samples were measured and recorded at 32th, 39th, 46th, and 53th using ion-selective electrode and digital pH meter.

Statistical analysis was done to find the relations between variables through using One-way ANOVA (Tukey test); a P-value < 0.05 was considered as statistically significant.

RESULTS

Table 1, Fig 1 shows mean values and standard deviations of the three materials during the 30 day period before solution treatment. Group1 (Fuji IX) and Group2 (Ketac nano) show high release of fluoride (90.097) and (64.819) at day 1 and sudden fall on day 2 then gradual constant decrease over time, the lowest value is in the thirty days which is (18.186) for Fuji IX, (10.711) for Ketac. While Group3 (Gradia x) shows lower release and leveling off thereafter without any sudden release or decrease in which the fluoride ion released at constant low level, the mean value is (25.100) in the first day and (1.63) in day thirty. The difference between the fluoride measurement of three restorative groups for mean values is statistically highly significant (P value <0.001).

Table 1: The Comparison between Mean Values and Standard deviation of Fluoride Ions Measurement of Three Studied Groups at Different Studied Periods

<table>
<thead>
<tr>
<th>Periods of measurement (days)</th>
<th>No.of samples</th>
<th>G1:Fuji IX GP Mean ± S.D</th>
<th>G2:Ketac nano Mean ± S.D</th>
<th>G3:Gradia X Mean ± S.D</th>
<th>P-values</th>
</tr>
</thead>
<tbody>
<tr>
<td>1st day</td>
<td>30</td>
<td>94.097 ± 0.698</td>
<td>64.819 ± 0.342</td>
<td>25.100 ± 0.197</td>
<td>&lt; 0.001</td>
</tr>
<tr>
<td>2nd day</td>
<td>30</td>
<td>58.828 ± 0.303</td>
<td>46.317 ± 0.474</td>
<td>17.396 ± 0.381</td>
<td>&lt; 0.001</td>
</tr>
<tr>
<td>7th day</td>
<td>30</td>
<td>45.895 ± 0.340</td>
<td>33.026 ± 0.293</td>
<td>9.008 ± 0.057</td>
<td>&lt; 0.001</td>
</tr>
<tr>
<td>14th day</td>
<td>30</td>
<td>32.350 ± 0.573</td>
<td>21.774 ± 0.589</td>
<td>4.499 ± 0.216</td>
<td>&lt; 0.001</td>
</tr>
<tr>
<td>30th day</td>
<td>30</td>
<td>18.186 ± 0.748</td>
<td>10.711 ± 0.852</td>
<td>1.633 ± 0.415</td>
<td>&lt; 0.001</td>
</tr>
</tbody>
</table>

Table 2, Figure 2, 3, 4 shows the mean values after treatment solutions, the mean value of materials were dropped and continues to decrease with no effect of fluoride recharge ability by de ionized water. After treatment with mouthwash, the highest value is in the 32nd day (26.768) for Fuji IX, (20.473) for Ketac N, (1.567) for Gradia but the mean value dropped quickly to a constant, low level at 39th and 46th days, and at day 53rd when refluoridation is administered on daily basis the rate increased significantly. While for Gradia X dropped sharply to a very low level at 39th and 46th days when treatment is done once a week which seems almost negligible. After treatment with toothpaste, the highest value is in the 32nd day which is (36.654) for Fuji IX, (26.768) for Ketac N and (4.160) for Gradia X but the mean value dropped to a constant, low level at 39th and 46th days, at day

Fig. 1 Mean levels of fluoride release in the first part of study.
53th when refluoridation is administered on daily basis the rate of fluoride ion release increased significantly for all three materials.

Table 2: Mean and standard deviation (SD) of fluoride release from the three restorative materials studied with different treatments.

<table>
<thead>
<tr>
<th>Periods of measurement (days)</th>
<th>Treatment</th>
<th>G1: Fuji IX GP Mean ± S.D</th>
<th>G2: Ketac nano Mean ± S.D</th>
<th>G3: Gradia X Mean ± S.D</th>
</tr>
</thead>
<tbody>
<tr>
<td>32th day</td>
<td>C</td>
<td>16.731 ± 0.129</td>
<td>8.746 ± 0.017</td>
<td>1.064 ± 0.015</td>
</tr>
<tr>
<td></td>
<td>MW</td>
<td>26.738 ± 0.016</td>
<td>20.473 ± 0.033</td>
<td>1.567 ± 0.024</td>
</tr>
<tr>
<td></td>
<td>TP</td>
<td>36.654 ± 0.016</td>
<td>26.768 ± 0.027</td>
<td>4.160 ± 0.021</td>
</tr>
<tr>
<td>39th day</td>
<td>C</td>
<td>10.829 ± 0.015</td>
<td>6.112 ± 0.020</td>
<td>0.790 ± 0.073</td>
</tr>
<tr>
<td></td>
<td>MW</td>
<td>9.596 ± 0.020</td>
<td>8.737 ± 0.022</td>
<td>0.880 ± 0.091</td>
</tr>
<tr>
<td></td>
<td>TP</td>
<td>16.858 ± 0.013</td>
<td>10.857 ± 0.014</td>
<td>1.268 ± 0.013</td>
</tr>
<tr>
<td>46th day</td>
<td>C</td>
<td>7.829 ± 0.015</td>
<td>4.112 ± 0.020</td>
<td>0.350 ± 0.108</td>
</tr>
<tr>
<td></td>
<td>MW</td>
<td>8.148 ± 0.024</td>
<td>6.737 ± 0.022</td>
<td>0.420 ± 0.078</td>
</tr>
<tr>
<td></td>
<td>TP</td>
<td>14.858 ± 0.013</td>
<td>9.357 ± 0.014</td>
<td>0.350 ± 0.108</td>
</tr>
<tr>
<td>53th day</td>
<td>C</td>
<td>4.731 ± 0.129</td>
<td>2.206 ± 0.195</td>
<td>0.120 ± 0.042</td>
</tr>
<tr>
<td></td>
<td>MW</td>
<td>22.738 ± 0.016</td>
<td>16.473 ± 0.033</td>
<td>0.367 ± 0.024</td>
</tr>
<tr>
<td></td>
<td>TP</td>
<td>32.654 ± 0.016</td>
<td>22.768 ± 0.027</td>
<td>1.860 ± 0.021</td>
</tr>
</tbody>
</table>

MW: Mouth wash, TP: Toothpaste, C: Control

Fig 2: Mean levels of fluoride release in control group treatment

Fig 3: Mean levels of fluoride release after control group exposure to mouthwash

Fig 4: Mean levels of fluoride release after exposure to toothpaste.

DISCUSSION

Fluoride release before treatment:

The release of fluoride from dental materials is affected by various intrinsic and extrinsic factors. The intrinsic factor is the composition of the restorative materials that, while the extrinsic factor is the storage medium. This study shows that Fuji IX GP released highest fluoride ions on day one after immersion, this result was supported by Cildir and Sandalli, Kiran and Hegde who stated that the high release of fluoride is likely to result from the initial burst effect of fluoride released from the glass particles. After day one, fluoride decreased quickly at day two and became stabilized and gradual diminished over time to a low-level long-term release on day seven and fourteen, with lower levels obtained on the 30th day, these results are close to and come in agreement with Cildir and Sandalli.

While Ketac nano presented a slight difference to Fuji IX GP; the differences are mainly related to the presence of nanoparticles in the composition of Ketac nano. On the day one, fluoride release from Ketac nanowas high but slightly lower than Fuji IX GP; this result comes in agreement with Hegde and Shetty, Dionysopoulos et al., who shows that this phenomenon could be explained by its low solubility, since it was shown by scanning electron microscopy that the surface morphology of the Ketac nano does not reveal voids, cracks and micro-porosities after immersion in saline. Ketac nano released the highest amounts of fluoride ions on the first day but continued to release fluoride consistently and at constant level for 30 days; this finding is in agreement to Dionysopoulos et al., Paschoal et al.,
While for Gradia Direct X released considerably less fluoride than the other two restorative materials. The release occurred over 30 days, but most of the release occurred within the first week, and leveling off thereafter without any sudden release or decrease in which the fluoride ion released at constant low level, this result comes in agreement with Chan et al., 14, Naoum et al., 15 who stated that fluoride released from Gradia X is not a function of the setting reaction but due to the passive leaching that may occur from the fluoride containing filler particles. The low release may be due to the low amount of fluoride incorporated in these materials as fillers.

Fluoride release after recharging:
There was a significant difference in the amount of fluoride re-released compared to control group throughout the experimental period. After exposure to fluoridated toothpaste, the initial fluoride ion release increased significantly for all materials, in which Fuji IX GP and Ketac nano exhibited significantly higher fluoride re-release than Gradia Direct X composite. But after exposure to fluoridated mouthwash, the initial fluoride ion release from Fuji IX GP and Ketac nano exhibited significantly high fluoride re-release than Gradia X which is very lower and almost negligible, in which the highest rate was achieved by all materials on the first day of refluoridation, but dropped quickly to a constant, low level within several days, at day 53 when refluoridation is administered on daily basis during the third week, the rate of fluoride ion release increased significantly for all materials, therefore frequent external application of fluoride is necessary to maintain the constant high fluoride release.

But this re-release was significantly lower than the fluoride rate released initially, this patterns were supported by Dysipoulos et al., Cildir and Sandalli, who demonstrated that fluoride release from refluoridated specimens, though very significant, is always lower than the initial fluoride release from the freshly mixed material, the reason behind is that fluoride re-release occur partly by washout of fluoride ions that are retained on the surface or in the pores of the restorative.

In the current study, higher recharge ability was observed when tooth paste was applied compared to using mouth wash and with increasing of exposure time, the recharge ability was increased this comes in agreements to Mousavianasab and Meyers, the reason was to the high viscosity and sticky nature of the toothpaste, which was difficult to wash off, may have remained trapped in pores in the specimens to release more fluoride on the 1st day after recharge immersion.

CONCLUSION
The three fluoride-containing dental materials used were able to release fluoride and exhibited different fluoride release patterns depending on their compositions.

The highest amount of fluoride release was from Fuji IX GP, followed by Ketac nano and Gradia direct X ranked the lowest.

Fuji IX GP conventional GICs and Ketac nano GICs demonstrated a “burst effect while Gradia X composite resin did not show a significant initial high fluoride ion release but maintained a low and relatively constant level of release.

After exposure to fluoridated mouthwash and fluoridated toothpaste, all materials were recharged and continued releasing fluoride, in which recharge ability from the toothpaste group was found to be more successful than the mouthwash group.

REFERENCE