

# Evaluation of Degree of Conversion, Flexural Strength, and Microhardness of a Novel Flowable Resin Composite

Yeni Bir Akışkan Rezın Kompozitin Dönüşüm Derecesi, Eğilme Dayanımı ve Mikro Sertliğinin Değerlendirilmesi

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## ABSTRACT

**Aim:** This *in vitro* study aimed to evaluate the degree of conversion, and to compare the flexural strength, and microhardness of two flowable resin composites with different filler ratio.

**Materials & Methods:** Two flowable composite resins were used in this study, ZENIFLOW (65% filler by volume) and Dynamic flow (60% filler by volume) (President, Germany). Disc-shaped specimens were prepared from each material using silicone molds (8 x 2). All specimens were polymerized with an LED curing unit (Valo Cordless, Ultradent, USA) at 1000 mW/cm<sup>2</sup> for 20 s. Polishing procedures were performed with 4-stage finishing discs (BISCO, USA). Vickers microhardness values (VHN) (n=5) and hardness ratio (HR) of these samples were determined from the top/bottom surfaces with a microhardness tester after being stored in distilled water at 37°C for 15 days. Degree of conversion (DC) was evaluated using Fourier Transform Infrared Spectroscopy (FTIR) (n=5). Rectangular specimens (25 x 2 x 2) were prepared and subjected to three-point bending test (n=10) to determine flexural strength (F<sub>s</sub>) and flexural modulus (F<sub>m</sub>). After the flexural strength evaluation, the fractured surfaces were examined by stereomicroscope (Leica MZ7.5, Germany) and Scanning Electron Microscope (SEM) (Zeiss EVO MA10, Germany). Independent sample t-test was used to compare the values between groups. The significance level was determined as p<0.05.

**Results:** There was no significant difference between the two resin composites with different filler ratios in terms of degree of conversion, microhardness, hardness ratio, flexural strength, and flexural modulus.

**Conclusion:** A 5% increase in the volume of filler content did not alter the degree of conversion and the tested mechanical properties of the resin composite.

**Keywords:** Degree of conversion, Flexural strength, Flowable resin composite, Microhardness, SEM.

## ÖZ

**Amaç:** Bu *in vitro* çalışmanın amacı, farklı doldurucu oranına sahip iki akışkan rezin kompozitin eğilme dayanımını ve mikrosertliğini karşılaştırmak, dönüşüm derecesini değerlendirmektir.

**Gereç ve Yöntemler:** Bu çalışmada ZENIFLOW (hacimce %65 doldurucu oranı) ve Dynamic flow (hacimce %60 doldurucu oranı) rezin kompozitler (President, Almanya) olmak üzere 2 adet akışkan kompozit rezin kullanıldı. Her bir materyalden silikon kalıplar (8x2) kullanılarak disk şeklinde örnekler hazırlandı. Tüm örnekler 20 saniye süreyle 1000 mW/cm<sup>2</sup> güçte LED ışık cihazı (Valo Cordless, Ultradent) ile polimerize edildi. Polisaj prosedürleri 4 aşamalı bitim diskleri (BISCO, ABD) ile gerçekleştirildi. Bu örneklerin Vickers mikrosertlik değerleri (VHN) (n=5) ve sertlik oranı (HR) alt/üst yüzeylerden mikrosertlik test cihazı ile 37°C'de distile suda 15 gün bekletildikten sonra belirlendi. Dönüşüm derecesi (DC), Fourier Dönüşümlü Kızılötesi Spektroskopisi (FTIR) kullanılarak değerlendirildi (n=5). Eğilme dayanımı (F<sub>s</sub>) ve elastisite modülünü (F<sub>m</sub>) belirlemek için dikdörtgen örnekler (25 x 2 x 2) hazırlandı ve üç nokta eğme testine (n=10) tabi tutuldu. Eğilme dayanımı değerlendirmesinin ardından oluşan kırık yüzeyler stereomikroskop (Leica MZ7.5, Almanya) ve Taramalı Elektron Mikroskobu (SEM) (Zeiss EVO MA10, Almanya) ile incelendi. Değerleri gruplara göre karşılaştırmak için bağımsız örneklem t-testi kullanıldı. Anlamlılık düzeyi p<0,05 olarak belirlendi.

**Bulgular:** Farklı doldurucu oranlarına sahip iki rezin kompozit arasında dönüşüm derecesi, mikrosertlik değeri, sertlik oranı, eğilme dayanımı ve elastisite modülü açısından anlamlı bir fark bulunmadı.

**Sonuç:** Doldurucu oranında hacimce %5'lik bir artış, dönüşüm derecesini ve rezin kompozitin test edilen mekanik özelliklerini değiştirmedi.

**Anahtar Kelimeler:** Akışkan rezin kompozit, Dönüşüm derecesi, Eğilme dayanımı, Mikrosertlik, SEM.

## INTRODUCTION

Resin composites have evolved into the material of choice for direct restorations in dentistry due to the improvements in their mechanical properties.<sup>1</sup> Although not all mechanical properties can be optimized in one material<sup>2,3</sup>, many parameters of resin composites such as fracture toughness<sup>4,5</sup>, flexural strength<sup>6,7</sup>, tensile strength<sup>8,9</sup>, and elastic modulus<sup>6,10</sup> have been investigated in many studies. Among these properties, flexural strength and flexural modulus are critical parameters because they affect the material's behavior under intraoral conditions.<sup>11</sup>

The development of flowable composites has emerged as a significant advancement in restorative dental materials. These materials are defined as low-viscosity resin composites derived from formulations with 20-25% lower filler loading compared to conventional composites. Their reduced viscosity allows for precise placement using injection syringes. First-generation flowable composites, characterized by a low elastic modulus, were primarily used as liners.<sup>12,13</sup> However, second-generation

flowable composites have been designed with improved mechanical properties and are now proposed for use in permanent restorations. Despite these advancements, concerns persist in the literature regarding their mechanical properties<sup>14,15</sup>, microleakage<sup>16,17</sup>, and post-operative sensitivity.<sup>18</sup> Various tests, including microhardness, flexural strength, and fracture toughness, have been utilized to evaluate the mechanical properties.<sup>6,14,19</sup>

The *in vitro* three-point flexural bending test is recommended by the ISO 4049:2019 specification for polymer-based materials and is widely used for comparative assessment. This makes it possible to compare mechanical properties with flexural strength (F<sub>s</sub>) and flexural modulus (F<sub>m</sub>) values. These mechanical properties of resin-based composites depend primarily on their microstructure and composition.<sup>11</sup> In addition, the resin content and the conversion from monomer to polymer structure affect the properties of the material.<sup>20</sup> It is believed that the adequate light polymerization of resin monomers is essential during the formation of a high-crosslinking polymer to achieve superior physical and mechanical properties in resin composites.<sup>21</sup> Fourier

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Transform Infrared Spectroscopy (FTIR) is one of the most commonly used methods for *in vitro* investigation of the degree of conversion (DC) of resin-based materials.<sup>22</sup> It is well known that the optimal DC or polymerization of resin-based materials is related to the characteristics of their physical, mechanical, and surface properties.<sup>6,23</sup>

The hardness of composite materials is a property that enables them to resist plastic deformation, penetration, indentation, and scratching. The microhardness of dental composites is commonly used to predict their abrasion resistance when used as restorations in functional areas.<sup>24</sup> Vickers microhardness (VHN) test is one of the most widely used methods for the assessment of microhardness.<sup>25</sup> In the past, many studies have found a positive correlation between DC values determined by microhardness or flexural strength and FTIR measurements.<sup>6,23,26,27</sup> Furthermore, the composition of resin composite materials, including the quantity of filler and the variety of monomers, is constantly updated<sup>28</sup>, and a variety of materials with different compositions are available on the market from various manufacturers. The composition of resin composites is known to undergo modifications which result in alterations to the physical, mechanical, and chemical properties of these materials.<sup>29-31</sup>

Considering the information provided, the objective of this study was to compare the degree of conversion, flexural strength, and Vickers microhardness values of a flowable resin composite and a novel flowable resin composite with a modified filler volume ratio. The null hypothesis was that there were no differences in the degree of conversion, microhardness, and flexural strength evaluation between the flowable resin composites with different filler ratios.

## MATERIAL AND METHODS

This *in vitro* study was conducted with two flowable resin composites (Dynamic Flow, President, Germany; ZENIFLOW, President, Germany) with different filler volume ratios of 60% and 65%, respectively. The compositions of the flowable resin composites are presented in Table 1.

**Table 1. The composition of the flowable resin composites was used in this study.**

| Resin Composite | Matrix                                                                                      | Filler                                     | Manufacturer                |
|-----------------|---------------------------------------------------------------------------------------------|--------------------------------------------|-----------------------------|
| Dynamic Flow    | Bis-GMA, UDMA, Ethoxylate Bis-GMA, TEGDMA, Trimethylolmethacrylate, Glycerol dimethacrylate | 60% (volume) barium alumino-boro-silicate. | President (Munich, Germany) |
| ZENIFLOW        | Bis-GMA, UDMA, Ethoxylate Bis-GMA, TEGDMA, Trimethylolmethacrylate, Glycerol dimethacrylate | 65% (volume) barium alumino-boro-silicate. | President (Munich, Germany) |

Abbreviations, Bis-GMA: bisphenol A-glycidyl methacrylate, UDMA: Urethane-dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate.

The quota sampling method was used to determine the number of samples used in the study, considering the budget and the sample sizes of other studies in the literature.<sup>6,32-34</sup> Sample preparations, test methods, and calculations of values utilized in this study were as follows:

### Degree of Conversion (DC)

The DC of the flowable resin composite samples was calculated using Fourier Transform Infrared Spectroscopy (FTIR-4000, JASCO, Japan). The spectral range and resolution were set at 400 to 4000  $\text{cm}^{-1}$  and 4  $\text{cm}^{-1}$ , respectively. Totally 10 disc-shaped (8 x 2 mm) samples were prepared with a silicone mold, glass slide, and a Mylar Strip (Hawe Transparent Strip, Kerr, Switzerland). Polymerization was completed using a polywave LED curing unit (Valo Cordless, Ultradent, USA) with an irradiance of 1000  $\text{mW}/\text{cm}^2$  for 20 s. Samples were stored in darkness at 37°C for 15 days. The samples (5 uncured and 5 cured, per material) were used for analysis and their absorbance peaks were recorded. 1608  $\text{cm}^{-1}$  (internal aromatic carbon double bond) and 1634  $\text{cm}^{-1}$  (methacrylate) peaks were selected for DC calculations. The DC was calculated by the following formula:

$$\left( 1 - \frac{\left( \frac{1634 \text{ cm}^{-1}}{1608 \text{ cm}^{-1}} \right)_{\text{cured}}}{\left( \frac{1634 \text{ cm}^{-1}}{1608 \text{ cm}^{-1}} \right)_{\text{uncured}}} \right) \times 100$$

### Flexural Strength ( $F_s$ ) and Flexural Modulus ( $F_M$ )

Twenty rectangular samples ( $n=10$ ) with dimensions of 25 x 2 x 2 mm were prepared in accordance with ISO 4049:2019 specification. Samples were polymerized at three points with an LED curing unit (Valo Cordless, Ultradent, USA) for 20 s. The top surfaces of the resin composite samples were polished with 200-grit paper. Following 15 days of storage (37°C) in darkness,  $F_s$  and  $F_M$  of the flowable resin composites were calculated through a three-point bending test using a universal test machine (AG-X, Shimadzu, Japan). The crosshead speed and supporting span were set at 1 mm/min and 20 mm, respectively. The maximum force (N) obtained from the testing machine and  $F_s$  (megapascal/MPa) and  $F_M$  (gigapascal/GPa) were calculated as follows:

$$F_s = 3FL / (2BH^2)$$

( $F$ : maximum load (N);  $L$ : distance between the supports (mm);  $B$ : width of the sample (mm);  $H$ : height (mm))

$$F_M = FL^3 / 4BH^3d$$

( $F$ : maximum load;  $L$ : distance between the supports;  $B$ : width of the sample,  $H$ : height of the sample,  $d$ : deflection (mm))

### Vickers Microhardness (VHN)

Totally 10 disc-shaped (8 x 2 mm) samples were prepared with a silicone mold, glass slide, and a Mylar Strip (Hawe Transparent Strip, Kerr, Switzerland). After polymerization with the Valo Cordless curing unit and finishing procedures with 4-step polishing discs (BISCO, USA), samples were stored in darkness at 37°C for 15 days. Three random indentations were performed in the center of the top and bottom surfaces of each sample using a microhardness tester (HNV-2, Shimadzu, Japan) with 0.49N/g load and dwell time of 15 s. The average of three hardness measurements were recorded, and Vickers microhardness values were calculated as follows:

$$VHN = 1.8544P / d^2$$

(VHN: Vickers micro-hardness,  $P$ : the indentation load,  $d$ : the length of the diagonal of the indentation)

The assesment of depth of cure was performed using the hardness ratio (HR), and the HR% was calculated using the following formula:

$$HR = \frac{\text{Vickers microhardness of the bottom surface}}{\text{Vickers microhardness of the top surface}} \times 100$$

### Stereo Microscope (SM) and Scanning Electron Microscope (SEM)

Following the three-point bending test, fractured surfaces of the samples ( $n=1$ ) were examined with a stereomicroscope (MZ7.5, Leica Microsystems, Germany) under x20 magnification, and Scanning Electron Microscope (SEM) (EVO MA10, Zeiss, Germany) under 10.00 kV and an approximate of 9 between 11 mm working distance. SEM images were obtained under x50, x100, x200, and x500 magnifications. Before SEM analysis, the samples were coated with a thin layer of gold at 20 mA for 180 s.

### Statistical Analysis

Data were analyzed with SPSS V23 (IBM, USA). Normal distribution was evaluated by Shapiro Wilk test. Independent samples t-test was used to compare the maximum force, flexural strength, flexural modulus, microhardness, and DC values according to composite groups. The significance level was set at  $p < 0.05$ .

## RESULTS

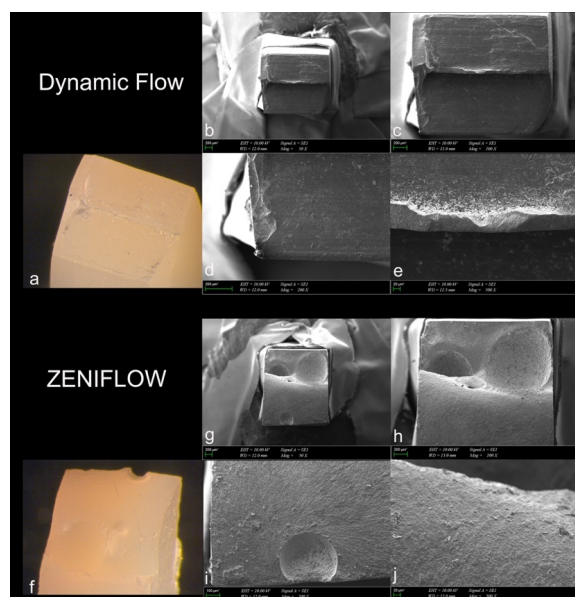
No statistically significant difference was found between ZENIFLOW and Dynamic Flow for DC evaluation ( $p=0.310$ ). Similarly, the mean values for maximum force,  $F_s$ ,  $F_m$  did not differ significantly according to the composite groups ( $p=0.051$ ;  $p=0.051$ ;  $p=0.232$ , respectively). No significant difference was obtained between the composites regarding top and bottom microhardness values ( $p=0.295$ ,  $p=0.117$ , respectively). Furthermore, the hardness ratio of the resin composites did not reveal a statistically significant difference ( $p=0.710$ ) (Table 2).

**Table 2.** Descriptive statistics of DC, maximum force,  $F_s$ ,  $F_m$ , bottom/top microhardness, and HR comparisons of resin composite materials.

| Properties            | ZENIFLOW (65% filler by Vol.) |                             | Dynamic Flow (60% filler by Vol.) |                          | Test St | P     |
|-----------------------|-------------------------------|-----------------------------|-----------------------------------|--------------------------|---------|-------|
|                       | Mean $\pm$ SD                 | Median (min-max)            | Mean $\pm$ SD                     | Median (min-max)         |         |       |
| DC                    | 71.01 $\pm$ 7.91              | 72.312<br>(59.237 - 78.534) | 65.10 $\pm$ 9.27                  | 64.543                   | 1.085   | 0.310 |
| Max. Force            | 27.3 $\pm$ 3.5                | 27.9<br>(21.5 - 31.6)       | 23.8 $\pm$ 4.1                    | 23.3<br>(18.2 - 31.3)    | 2.087   | 0.051 |
| FS                    | 102.3 $\pm$ 13.1              | 104.8<br>(80.6 - 118.4)     | 89.1 $\pm$ 15.3                   | 87.5<br>(68.1 - 117.2)   | 2.087   | 0.051 |
| EM                    | 2.8 $\pm$ 0.5                 | 2.7<br>(2.2 - 3.5)          | 2.5 $\pm$ 0.4                     | 2.5<br>(1.7 - 3.1)       | 1.238   | 0.232 |
| VHN of top surface    | 46.98 $\pm$ 10.11             | 45.63<br>(35.13 - 61.4)     | 41.49 $\pm$ 4.22                  | 39.7<br>(35.4 - 47.23)   | 1.121   | 0.295 |
| VHN of bottom surface | 36.41 $\pm$ 2.38              | 36.2<br>(34.3 - 39.66)      | 32.05 $\pm$ 5.00                  | 33<br>(25.5 - 36.9)      | 1.759   | 0.117 |
| Hardness ratio (%)    | 71.01 $\pm$ 7.91              | 86.92<br>(55.48 - 97.62)    | 65.10 $\pm$ 9.27                  | 78.12<br>(67.67 - 89.45) | 0.385   | 0.710 |

\*Two independent sample t-tests, DC: degree of conversion, Max. Force: Maximum Force (N),  $F_s$ : Flexural Strength (MPa),  $F_m$ : Flexural Modulus (GPa), VHN: Vickers Microhardness, Mean  $\pm$  SD: Mean  $\pm$  Standard Deviation, Median (Min-Max): Median (Minimum-Maximum), Test St: Test Statistics

Stereomicroscopy did not show any difference in the macro appearance of the fractured surfaces between the groups. The surface structures remained unaltered as the matrix and filler structures were identical. The images revealed void formation in the ZENIFLOW resin composite group. The fracture patterns observed in the composites were distinct, attributable to the formation of voids (Fig. 1).



**Figure 1.** a. Stereomicroscopy image of Dynamic Flow (20 $\times$ ). b-e.

SEM images of Dynamic Flow (50 $\times$ , 100 $\times$ , 200 $\times$ , and 500 $\times$  magnifications, respectively). f. Stereomicroscopy image of the ZENIFLOW (20 $\times$ ). g-j. SEM images of ZENIFLOW (50 $\times$ , 100 $\times$ , 200 $\times$ , and 500 $\times$  magnifications, respectively).

## DISCUSSION

Due to their esthetic appearance and improved mechanical properties, resin composites are increasingly used in dentistry. Dental resin composites are composed of two main components: organic resin matrix and organic/inorganic fillers. Organic resins consist of a mixture of multifunctional monomers and initiators, while the fillers can be organic or inorganic with varying sizes, shapes, and functions.<sup>35</sup>

Despite advances in resin composite technology, the most significant developments have been achieved in the ratio, structure, shape, and functionality of fillers.<sup>36-38</sup> The fillers used in the dental resin composites are usually inorganic particles. The classification of composites is dependent upon the size of the fillers such as macro, micro, and nano. However, the demarcation between micro-hybrid and nano-hybrid composites is not readily apparent, given that both categories contain micro and nanoparticles and exhibit a comparable particle size distribution.<sup>39</sup> In this study, a micro-hybrid and flowable resin composite, as defined by the manufacturer, and a newly introduced composite with an identical composition were evaluated. However, the micro-hybrid flowable resin composite (Dynamic Flow) had a filler content of 60%, while the novel composite (ZENIFLOW) had a filler content of 65% (by Vol.).

Several research findings indicate that the  $F_m$  exhibits an exponential increase with an increase in filler fraction for dental resin composites.<sup>40-43</sup> Mirică et al.<sup>41</sup> stated that the percentage ratio of inorganic filler was significantly correlated with the mechanical properties of flowable resin composites. Furthermore, the presence of hybrid inorganic fillers in the composition of some materials was observed to result in enhanced mechanical properties. In addition, previous studies stated that the filler ratio of dental resin composites is directly proportional to their microhardness.<sup>35,43</sup> On the contrary, the present study revealed no significant difference in the evaluated mechanical properties between composite materials with varying filler ratios. Similarly, an examination of the fractured surfaces after the bending tests revealed no discernible structural difference between the two composite groups. The observed differences in fracture type may be attributed to the formation of voids, which could result in more rounded fracture morphology. Moreover, the lack of differences observed in this study might be attributed to the fact that previous studies have compared composites from different manufacturers with varying filler ratios, whereas in this study, two identical composites were evaluated, differing only in their filler ratios. In the two composite groups, the microhardness of the bottom surfaces was lower than the top surfaces. This can be explained as the higher amount of filler particle content of the resin-based composite explains the reduced light transmittance and the consequential lower DC at the bottom of the samples.<sup>44</sup> It is also known that even if other compositions were identical, mechanical properties are affected by many structures in the resin composite composition and changes of conditions during production, and this is inherently intricate.<sup>36,38</sup> In the current study, a 5% change in the filler ratio in the same composition did not result in any significant change in the  $F_s$ ,  $F_m$ , and VHN values of micro-hybrid flowable resin composite. Likewise, the DC of these resin composites was not significantly different. In addition, a previous study found that the incorporation of the fillers can restrict the mobility of the monomers and radicals, leading to a decreased conversion.<sup>45</sup> However, the DC was not different between the two composites with 65% and 60% filler volume ratios in the current study. Considering the observed results in the evaluated mechanical properties and DC of the composites, the null hypothesis proposed in this study was accepted. Several studies revealed that there is a direct relationship and proportionality between the DC and the mentioned mechanical properties<sup>27,46-48</sup>, and this finding aligns with the results of the current study. Furthermore, de Mendonça et al.<sup>48</sup> claimed that the mechanical properties of light-cured materials may be affected by inadequate polymerization. In this study, all samples were polymerized with a polywave LED curing unit with an irradiance of 1000 mW/cm<sup>2</sup> for 20 s. Since adequate irradiance, power, and time were utilized for all samples in this study, differences may not have been observed in the DC and mechanical properties. It should be noted that the present study is limited by the fact that curing units with different irradiance or different polymerization durations were not evaluated. It would be beneficial for further research to include the long-term evaluation of the resin composites under different oral conditions using artificial aging methods.

## CONCLUSION

Under the limitations of this *in vitro* study, The mechanical properties (flexural strength, flexural modulus, microhardness) and degree of conversion of flowable resin composites with 60% and 65% filler content by volume, which have the same matrix structure and filler content, are similar.

**Değerlendirme / Peer-Review**

İki Dış Hakem / Çift Taraflı Körleme

**Etik Beyan / Ethical statement**

Bu makale "2022 RDD Kış Sempozyumu ve Anabilim Dalları Toplantısı"nda poster olarak sunulan ancak tam metni yayımlanmayan "Deneysel Akışkan Rezin Kompozitin Bükülme Dayanımı ve Elastik Modülünün Değerlendirilmesi" adlı tebliğin içeriği geliştirilerek ve kısmen değiştirilerek üretilmiş halidir.

Bu çalışmanın hazırlanma sürecinde bilimsel ve etik ilkelere uyulduğu ve yararlanılan tüm çalışmaların kaynakçada belirtildiği beyan olunur.

This article is a partially modified and improved version of the paper titled "Evaluation of Flexural Strength and Elastic Modulus of Experimental Flowable Resin Composite" which was presented as a poster at the "2022 RDD Winter Symposium and Department Meeting" but the full text was not published.

It is declared that during the preparation process of this study, scientific and ethical principles were followed and all the studies benefited are stated in the bibliography.

**Benzerlik Taraması / Similarity scan**

Yapıldı - ithenticate

**Etik Bildirim / Ethical statement**

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**Çıkar Çatışması / Conflict of interest**

Çıkar çatışması beyan edilmemiştir.

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Veri Toplanması | Data Acquisition: BDK (%25), SÖ (%25), NZK (%25), AAŞ (%25)

Veri Analizi | Data Analysis: BDK (%25), SÖ (%25), NZK (%25), AAŞ (%25)

Makalenin Yazımı | Writing up: BDK (%50), SÖ (%30), AAŞ (%20)

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