### 2.2. Experimental Methods

The wires were analyzed by scanning electron microscopy (SEM) and X-ray energy dispersive spectroscopy (EDS). The microstructure of the wires surface was studied by means of a Zeiss EVO MA-15 scanning electron microscope with a LaB6 cathode on polished cross-section samples. The chemical composition was determined by EDS using an Oxford Instruments INCA Energy system. The qualitative and quantitative analyses were carried out at an accelerating voltage of 20 kV, an optimal condition for these samples.

## 3. Results and Discussion

Analyzing the composition of the archwires studied, we compared the data obtained about the new as-received archwires with the data in the scientific literature and found a good agreement. The EDS analysis of the elemental composition of the stainless steel archwires confirmed that made by [8, 9]. In the nickel-titanium (Ni-Ti) archwires, the percentage ratios of nickel and titanium were the same as those quoted by [9, 10]. For the thermally-activated (TMA) archwires, the analysis again confirmed the data of [11] for the chemical composition of the titanium-molybdenum alloy wires.

In Ni-Ti archwires, we observed an increase in the titanium content by + 0.17%, and a decrease of the nickel content by 0.17%, which we attribited to the experimental error (Table 3). The deviations are without a statistically significant difference, and are within the values cited in the literature [12, 13]. In the other types of arches – beta-titanium (C) and copper-nickel-titanium (D) – we observed insignificant changes in the chemical composition. In the stainless steel archwires (A), we found a significant increase in the iron content (+0.59%, Table 2), which is likely due to variations in the composition of the different batches of archwires, or to deposits on the archwires caused by water vapor in the autoclave after drying [14]. Table 2 shows the results for the chemical composition of a steel orthodontic archwire (A), unused and unused sterilized (autoclaved), as obtained by the EDS analysis. The data are compared with sources from the literature [9, 13].

**Table 2:** Elemental content of as-received and of sterilized type A wires.

| *Elements, weight %* | *Si* | *Cr* | *Mn* | *Fe* | *Ni* | *Total* |
|---|---|---|---|---|---|---|
| Industrial data (information) | ~1 | 18÷20 | ~2 | ~ 71 | 8÷11 | |
| A0 (as-received) Initial components content | 0.82 | 19.76 | 1.54 | 69.08 | 8.80 | 100 |
| A1 Mean components content after sterilization process | 0.82 | 19.70 | 1.44 | 69.67 | 8.38 | 100 |
| | 0 | -0.06 | -0.1 | +0.59 | -0.42 | |

The study of the chemical composition of the type A wires (as-received and as-received sterilized) demonstrated no statistically significant difference. The changes seen in the amount of iron with a higher average value of 0.59%, and a lower average value of amount of nickel of 0.42%, should not be expected to affect the surface and the properties of the material. Figure 1 shows the microstructures of the surfaces of the unused and unused sterilized (A) archwires respectively, as studied by a scanning electron microscope.

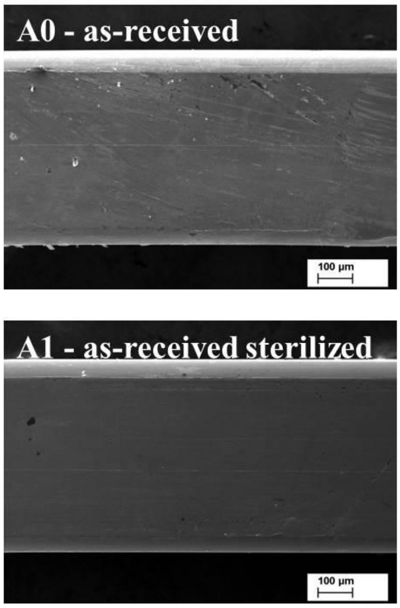


**Figure 1:** Scanning electron images of the surface of SS steel archwires, A0 - as-received A1 - as-received sterilized (autoclaved).

The above measurements did not establish any changes on the surface structure of the archwires. Processes of corrosion or increased roughness were not observed. The practically negligible differences that we found were likely due to the fact that the studies were carried out at different points, the alloy not being completely homogeneous, or to the equipment's measurement error. We can note that the surface of the steel archwires was the smoothest compared to the other archwires tested. The data obtained by the EDS analysis about the ratio of the elements in the nickel-titanium orthodontic archwires before and after autoclaving are provided in Table 3, together with the data quoted in the references.

**Table 3:** Elemental content of as-received and sterilized type B wires

| *Elements, weight %* | *Ni* | *Ti* | *Total* |
|---|---|---|---|
| Industrial data (information) | 54÷55 % | 43÷44 % | |
| B0 (as-received) Initial components Content | 54.56 | 45.44 | 100 |
| B1 - Mean components content after sterilization process | 54.73 | 45.27 | 100 |
| | +0.17 | -0.17 | |

The data about the Ni-Ti archwires made it obvious that the changes in the chemical composition were 0.17 %, while there were no visible changes on the surface, whose images are shown in Figure 2.

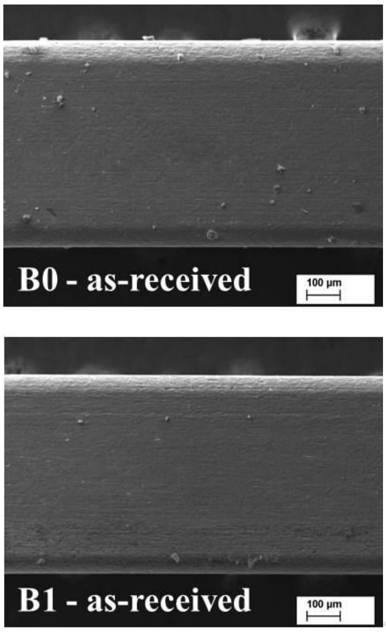


**Figure** 2: Scanning electron images of nickel-titanium archwires' surface of, B0 - as-received and B1 - as-received sterilized (autoclaved).

The SEM images showed surface irregularities. According to Pernier et al. [7] and Van Hoogstraten et al. [15], the higher degree of roughness can be explained by the fact that the sterilization removes residues and oily traces that may have remained in the archwires after their production. The results of the EDS analysis of as-received and as-received sterilized orthodontic archwires of beta-titanium alloy (C) established minor changes in the chemical composition, namely, +0.10% to -0.15%, which is within the existing method's error (Table 4). No signs of corrosion could be seen on surface (Figure 3).

**Table 4:** Elemental content of as-received and sterilized type C wires.

| *Elements, weight %* | *Ti* | *Zr* | *Mo* | *Sn* | *Total* |
|---|---|---|---|---|---|
| Industrial data (information) | ~79 % | ~6 % | ~11 % | ~4 % | |
| C0 (as-received) Initial components content | 76.80 | 6.74 | 11.75 | 4.71 | *100* |
| C1 – Mean components content after sterilization process | 76.84 | 6.59 | 11.75 | 4.81 | 100 |
| | +0.04 | -0.15 | 0 | +0.10 | |

The thermally-activated archwires are characterized by structural transformations during changes in the temperature (martensite-austenite). Bearing this in mind, we performed X-ray structural analysis for these orthodontic archwires (Figure 4) on the as-received and as-received autoclaved archwires with copper content (D1). The analysis showed that after the sterilization processes there were no structural changes in the thermally-activated archwires.


**Figure 3:** Scanning electronic images of the surface of TMA archwire, C0 - as-received and C1 - as-received sterilized (autoclaved).

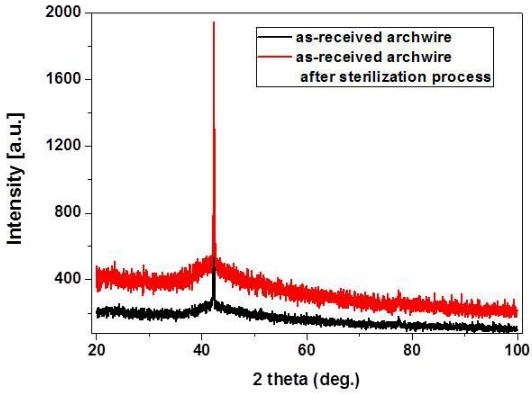


**Figure 4:** X-ray spectra of thermally-activateed archwires with copper content: as-received (black) and as-received (autoclaved) (red).

In these, as well as in the other orthodontic archwires studied, no significant changes on the surface were observed due to the autoclaving process (Figure 5). The differences established in the chemical composition were within the experimental error, while no corrosion processes were seen on the surface (Table 5).

Bearing in mind the above, we believe we can summarize the results by concluding that the autoclaving processes did not affect the crystal structure and the parameters of the archwires investigated. The analyses carried out by SEM and by EDS did not reveal statistically significant changes in the chemical composition of the autoclaved archwires. A possible reason for the variations observed in the chemical composition was the complexity of the production procedures on the one hand, and on the other, the difference in the properties of the archwires within separate batches. This finding strongly limits the consistency of the experimental data [7].

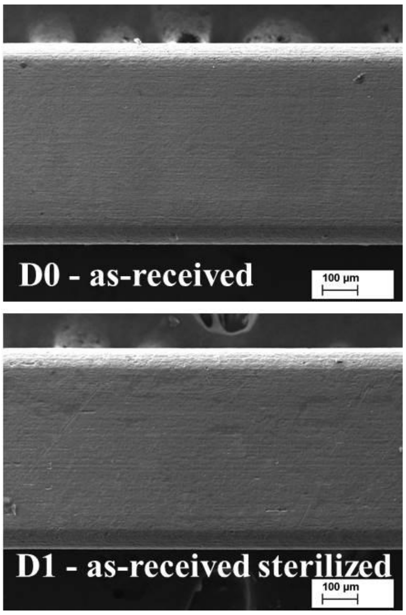


**Figure 5:** Scanning electronic images of the surface of thermally-activated orthodontic archwire with copper content, D0 - as-received D1 - as-received autoclaved.

**Table 5:** Elemental content of as-received and sterilized type D wires

| *Elements, weight %* | *Ti* | *Ni* | *Cu* | *Cr* | *Total* |
|---|---|---|---|---|---|
| Industrial data (information) | ~43 % | ~50 % | ~6.5 % | ~0.5% | |
| D0 (as-received) Initial components content | 45.58 | 48.39 | 5.56 | 0.47 | 100 |
| D1- Mean components content after sterilization process | 45.60 | 48.33 | 5.66 | 0.41 | 100 |
| | +0.02 | -0.06 | +0.10 | -0.06 | |

## 4. Conclusions

The lack of statistically significant differences established in the study of new and autoclaved orthodontic archwires gives us reason to conclude that the orthodontic arches can be sterilized because the autoclaving processes do not affect their properties and the orthodontists could thus ensure the maximum safety of their patients.

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