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ORIGINAL ARTICLE

Potential use of Friction Welding for Fabricating Semi-Biodegradable Bone Screws

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ABSTRACT – Certain surgical interventions, such as fractures, may require implants that have a combination of external and internal parts made of inert and biodegradable biomaterials, respectively. This implant design can be fabricated using specific fabrication methods such as friction welding that are able to efficiently combine two biomaterials. This study reports the utilization of the direct friction welding technique for the fabrication of semi-biodegradable bone screws using two parent metals of low carbon steel and austenitic stainless steel 202. The welding parameters were optimized to obtain welded joints for bone screw fabrication. The mechanical properties of metals that have been welded were identified through tensile and three-point bending analyses. The corrosion test was then conducted on the welded metals through the measurement of corrosion rate, changes in pH value, morphology visualization, and element release, while the cytotoxicity effect was evaluated through a cell viability test. Screw implant materials with a diameter of up to 12.7 mm were successfully fabricated using a continuous friction drive welding machine at 4000 rpm and 24.5 MPa hydraulic pressure. The results of mechanical testing show that the tensile strength of weld joints decreased by 3.6% from low carbon steel and 20.4% from stainless steel. Fractures were observed at the welding interface after being subjected to flexural testing. The pH value of the Saline solution decreased from 7.13±0.06 to 6.73±0.06 after the welded metals were immersed for up to 8 weeks. Evaluation of the surface morphology in all welding zones at week eight samples obtained that almost all types of corrosion that occurred were uniform corrosion, except in the PZ zone where galvanic corrosion was formed. The concentrations of Cr and Ni ions (ppm level) were very low, namely 0.058 ppm for Cr and 0.199 ppm for Ni. The viability of human fibroblast cells was maintained at higher than 75% viability after the cell incubation at 1, 3, and 5 days with different parts of welded joints.

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INTRODUCTION

Medical devices, whether used temporarily or permanently, both internally and externally used on the human body, have become more complex and sophisticated. As a result, many manufacturing and medical industries have utilized assembly (joining) methods to fabricate medical devices, where one of the commonly used processes is a welding technique [1]. A friction welding produces joints of exceptional quality [2] with little or no post-welding [3]. Despite the high quality of the joints, there are a number of advantages to friction welding, including low heat input, a small heat-affected zone (HAZ), and low residual stress and distortion [4, 5]. Besides, friction welding can also join dissimilar materials with good results [6, 7]. In the last several years, the joint of dissimilar metal joints have been amply used in academia and industry as it can join materials with different properties, is inexpensive, has high corrosion resistance, and has good biocompatibility to be used in the biomedical industry [8].

There are two types of friction welding; continuous-drive friction welding and inertia-friction welding [9, 10]. In this study, continuous-drive friction welding was used, where one component remains stationary, and the other rotates at a constant speed, as in Figure 1 [10]. There are several relationships between important parameters, including pressure and friction time, pressure and forging time, and rotational speed, see Figure 1(b) [3, 5-7, 10-14]. At the same time, inertia-friction welding involves a flywheel that stores energy which is ready to be released to unite the two components while applying axial pressure [10]. The purpose of this study was to fabricate semi-biodegradable bone screws for the biomedical industry using continuous-drive friction welding. The optimal conditions for producing high-quality friction welds on each material have not been detailed. The process of manufacturing small-dimensional semi-biodegradable bone screws will require smaller welding parameters than general welding. In addition, those parameters are associated with atomic diffusion between two parent materials. Therefore, extra precautions need to be considered on poisonous or toxic materials so that their atoms would not diffuse too far into a metal that will be implanted into the human body, according to Nasution et al. [5].

In the previous study, Ahmed Ghias et al. analyzed the mechanism of friction welding parameters using low carbon steel-stainless steel and aluminum-copper materials. While using these welding parameters, diffusion and recrystallization

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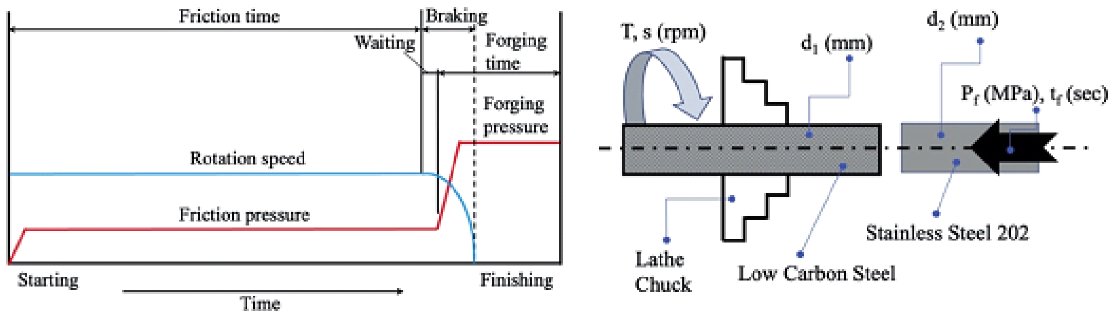
are important factors in friction welding low carbon steel-stainless steel. While cold working and upsetting seem to be the predominant mechanism in aluminum-copper welding [15]. Khidhir and Baban investigated the effects of frictional welding parameters on the microstructure and mechanical properties of AISI 1045 medium carbon steel and AISI 316L austenitic stainless steel [16]. James and Sudhish discussed the properties of friction welded joints with and without interlayer on different welding parameters between austenitic stainless steel 304 and medium carbon steel AISI 1040 [17]. Hong Ma et al. investigated the potential of a post-weld heat treatment process to produce the joint with desirable mechanical properties of carbon steels to stainless steels [18]. Nasution et al. carried out the development of metal bone pins using friction welding to connect pure iron-stainless steel 316L [5]. Paventhan et al. observed the behavior of mechanical properties (fatigue) of friction welded joints between medium carbon steel and austenitic stainless steel [19]. This article discusses the designing and manufacturing friction welding machines with small welding parameters, taking into account the size of the commonly used bone screws. In this study, semi-biodegradable bone screws made of low carbon steel and 202 stainless steel (SS 202) were fabricated using a friction welding method. The parent metals' chemical compositions were determined with an energy dispersive X-ray (EDS) instrument. At the same time, the welded metals were subjected to mechanical and corrosion tests. The biocompatibility of the welded metals was also assessed with human skin fibroblast cells through a cell viability measurement.



(a) (b)

Figure 1. (a) Parameters of continuous-drive friction welding and (b) equal section parts used in the experiments.

MATERIALS AND METHODS

Sample Preparation

Based on previous research on the manufacture of bone screws material using a friction welding process by connecting pure iron and 316L stainless steel [20], a similar method was utilized in this study where materials that are close were chosen to be the parent metals; low carbon steel and SS202. Each steel bar has a length of 70 mm and a diameter of 8 mm. Hereinafter, the steel bars were polished with #1000-grit abrasive paper and cleaned with ethanol. Energy dispersive X-ray (EDS, Hitachi TM3000, Japan) analysis was used to determine the chemical composition of the parent metals.

The polished rods were then installed on a welding machine chuck, as illustrated in Figure 2, which is a parameter of the method of friction welding used. By considering the dimensions of implant material, such as internal fixator (bone screw) [21] and external fixator (Schanz screw), as well as preliminary works [5, 20], the diameter of screws was set at 12.7 mm (max.) by using a rotary speed of 4000 rpm and hydraulic pressure of 25 MPa.

Table 1. Nominal chemical composition of the parent metals (wt.%).

Materials	C	Mn	Si	Cu	Mg	Zn	Cr	Ni	Mo	S	P	Fe
SS202	0.150	9.390	0.660	0.450	-	-	13.650	3.624	0.120	-	-	Bal.
Low carbon steel	0.202	0.648	0.449	-	-	-	0.325	-	-	0.016	0.019	Bal.

Mechanical Test

The welded metal and the parent metals were fabricated as tensile test specimens for small specimens following the ASTM E8 standard [22] with measuring gauge lengths of 30.0 ± 0.1 mm and a diameter of 6.0 ± 0.1 mm. The quality and characteristics of welded joints were evaluated using a universal tensile machine (Controlab-TN 20 MD, France) with a loading rate of 1.5 kN/min. A three-point bending test was then conducted on the welded metal and the parent metals following the ASTM E 290-14 standard [23] to identify the metal's flexural strength by applying a lateral load.

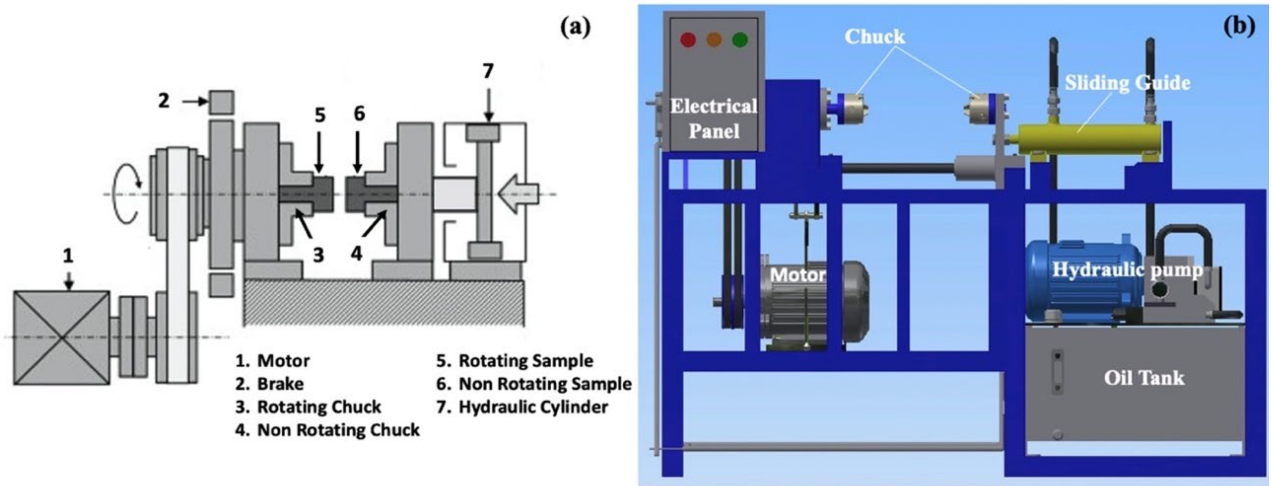


Figure 2. (a) Schematic of continuous-drive friction welding machine, and (b) assembly of friction welded machine used in the present study.

Biocorrosion Test

The corrosion test was carried out through an immersion procedure in a saline solution (NaCl 0.9%), 50 ml for each sample, following the ASTM G31 standard [24]. The welded metal was immersed in a water bath at 37 ± 1°C for 2, 4, 6, and 8 weeks, with five replications for each group. Then the sample was removed from the immersion solution, rinsed with distilled water and alcohol, and followed by a drying process at room temperature. Weight measurements were carried out on samples before and after immersion. The degradation rate is calculated based on the formula: CR = m/St [25], where CR denotes the corrosion rate (g cm^{-2} day^{-1}), m denotes the weight of the specimen prior to immersion (g), S denotes the specimen's surface area, and t denotes the time of immersion (day). The weight of the welded metals was determined prior to and following the immersion procedure using a 0.1 µg precision weighing balance.

Observations of changes in the surface morphology of the specimens after immersion were characterized by a stereo optical microscope (Motic Images Plus 2.0, China), where the morphology results were analyzed using Software Image J-NIH (Image J, National Institutes of Health, USA). The pH of the saline solution was determined prior to and following the immersion procedure at five replications. Following the immersion procedure, an atomic absorption spectrophotometer (AAS, AA-7000 Series, Shimadzu, Japan) was used to determine the ion concentration using a saline solution (20 mL).

Cell Viability Test

To uncover the biocompatibility of new materials for orthopaedic applications, a cell viability test is an indicator in the bio-evaluation to assess the response of physiological cells towards the new materials. Therefore, the cell viability test was conducted on the welded metals through an indirect contact approach with normal human skin fibroblast cells (HSF 1184, ECACC, UK) following ISO 10993-5 standard [26]. The indirect contact approach is intended to evaluate the cytotoxicity of the degraded materials. To distinguish between distinct regions of welded metals, the metals were divided into five zones: (1) undeformed zone of the low carbon steel (UZ-1), (2) undeformed zone of the SS202 (UZ-2), (3) partially deformed zone of the low carbon steel (PDZ-1), (4) partially deformed zone of the SS202 (PDZ-2), and (5) plasticized zone (PZ) [4, 5, 27]. Each zone was cut into a dimension of 4×4×2 mm. Prior to the cell viability test, the welded metals were ultraviolet (UV) sterilized for 15 minutes on each surface.

The cell media were prepared with a combination of Dulbecco Modified Eagle's Medium (DMEM) and fetal bovine serum (FBS) ratio of 10:1. Penicillin streptomycin (Pen Strep) was then incorporated into the media at 1% v/v for disinfection. The cells were cultured in the media with a supplement of 5% CO_2 at 37 ºC and 95% humidity. The sterilized specimens were treated with 2×10^5 cells/mL HSF for 1, 3, and 5 days in 24-wells and further incubated at 37 ºC, 95% humidity, and 5% CO_2.

At each specified time point, the cells were washed with 100 µL of phosphate buffer saline (PBS), and the medium in each well was replaced with 100 µL of 3-(4,5-Dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide (MTT) solution. Further incubation was performed for 4 hours, followed by the replacement of MTT solution with 150 µL of dimethyl sulfoxide (DMSO). Finally, absorbance values were determined using a microplate reader (BioTek808, USA) at a wavelength of 562 nm. Statistical analysis was performed to determine whether the triplicate measurements had a significant difference at a p-value<0.05 using the Student t-test and Tukey's comparison method.

RESULTS AND DISCUSSION

Experimental Setup

The result of the design of a friction welding machine is obtained by an electric motor with a power of 3 kW and 4114 rpm rotation so that it is sufficient for the torque capacity of steel rods during friction welding in diameters up to 12.7 mm. The hydraulic pressure and braking time was 24.5 MPa and 0.24 seconds, respectively. The assembly of friction welded machine components for the experimental part of the study is shown in Figure 2(a) and 2(b). The parent metals' chemical compositions are recorded in Table 1. The elements of Cu, Ni, and Mo were recorded in the SS202, where these elements are responsible for increased corrosion resistance, and it is reported that Cu elements have antibacterial abilities [28].

Mechanical Properties

The mechanical properties of the parent metals and the joints are presented in Table 2. The average tensile strength is 847.24 ± 13.99 MPa and the yield strength is 800.67 ± 14.79 MPa. The results of friction welding were obtained at the optimum welding parameters using a rotating speed of 4000 rpm, a friction pressure of 0.98 MPa, a forging pressure of 4.90 MPa, and a friction time of 8 s. The tensile strength of SS202 and low carbon steel were 20.4% and 3.6% decreased, respectively, as referred to the welded metals. The friction welding process is not always subjected to high strength properties but also could produce a soft zone in the weld joint [7, 29]. Therefore, in this study, the strength reduction is majorly contributed by the formation of a soft zone when both SS202 and low carbon steel were welded. According to the tensile test results, the fracture zone occurred between 1-3 mm from the welding interface (WI), near the low carbon steel zone (in Figure 3).

The average flexural strength of the welded metals caused by the lateral loads was measured to be 717.99±74.91 N where the fractured happened at the WI. This flexural strength shows a considerable decrease in strength and shows ductility of almost zero [13] based on the analysis on the test graph. Fauzi et al. mentioned that flexural strength could provide information on the strength of metal bonding from the quality of welded joints [30]. The weakness of friction welded joints can be influenced by several factors, including intermetallic brittleness, strain hardening, and residual stresses near the interface [31]. The weaknesses of the weld joints can also be interrelated with non-bonded areas and short friction times, resulting in inhomogeneous heating on the metal surface [27]. However, the friction times that exceed the optimum values will reduce productivity and increase material consumption [27]. At the same time, Lucas found that in mild steel, due to the combination of a long friction time and a low heating pressure, the ductility is low [32].

The maximum flexural strength in friction welded joints can be accomplished with high rotational speeds, where this relation is correlated with heat input [31]. The heat input values are calculated based on the combination of forging pressure, friction time, and burn-off length. In the previous literature [5], heat input greater than 500 J/s is classified as high heat input. In this study, the used welding parameters cause the projection of 0.3 J/s heat input, thus being categorized as the low heat input. The low heat input has less capability to provide energy for atomic rearrangement, thus reducing the possibility of atomic diffusion at the WI.

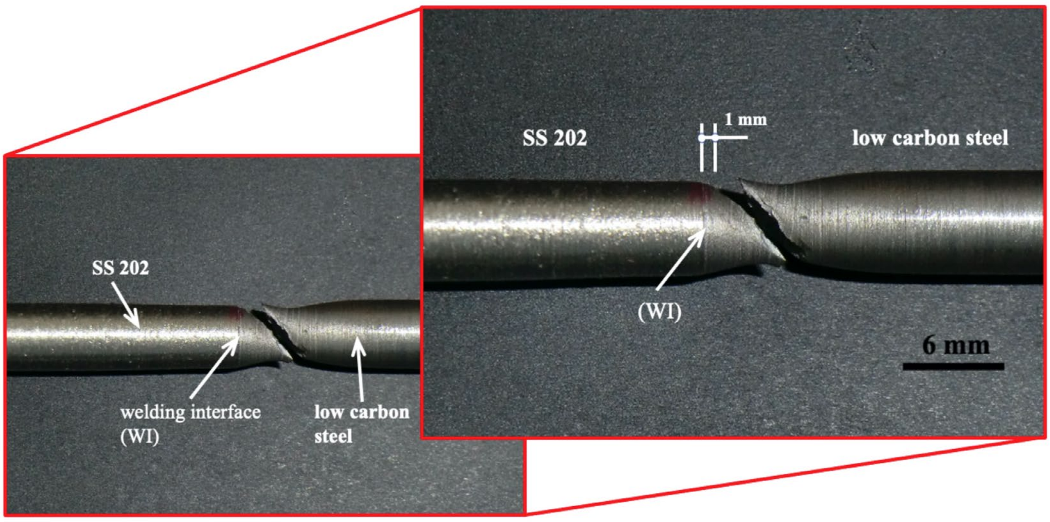


Figure 3. Determination of the fracture zone of one of the tensile test specimens.

Table 2. Mechanical properties of parent metals and friction welded joints.

Materials	Tensile strength		Flexural strength
	Yield strength (MPa)	Tensile strength (MPa)	Lateral load (N)
SS202	902.92 ± 24.50	1,064.60 ± 29.57	3782.20 ± 55.57
Low carbon Steel	851.82 ± 20.70	879.31 ± 24.74	2,403.98 ± 45.59
Friction Welding	800.67 ± 14.79	847.24 ± 13.99	717.99 ± 74.91

Corrosion Properties

The immersion procedure in the saline water at 2, 4, 6, and 8 weeks timepoints produced a decrement in corrosion rate curves (Figure 4). A high corrosion rate occurred in the second week, where the rate began to slow down following the next time point. The corrosion rate was slowed by the formation of a layer on the surface of the specimen; this layer is known as the passive layer [33]. In this study, the passive layer was formed in the UZ-1, PDZ-1, and PZ zones. Chloride ions, on the other hand, may dislodge the passivation layer at some weak points or easily penetrate the oxide layer to reach the Fe substrate [33]. A similar phenomenon was observed by Nasution et al., where the iron hydroxide layer formed but was gradually eroded away by day 25 [20]. The pH value of the saline solution is shown in Figure 5. There was a decrease in the pH value from 7.13 ± 0.06 to 6.73 ± 0.06 after the welded metals were immersed for eight weeks. According to Chen et al., the decrease in pH was caused by the iron chloride being hydrolyzed in the confined space beneath the oxide layer [33]. Figure 6 shows the surface morphology (week 8) and the surface roughness values of each sample. Almost all of the types of corrosion that occur in the welding area are uniform corrosion. The same thing was also found by Zhu et al. on immersion of the iron stents for approximately one month in simulated body fluids (SBF) [34]. In the UZ-2 and PDZ-2 zones, there is no rough surface due to the corrosion process. The roughest surface occurs in the PZ zone, followed by the PDZ-1 zone and the UZ-1 zone. Whereas in the PZ zone, the corrosion that is formed is a form of galvanic corrosion due to the presence of two different metals or alloys in contact with each other or touching in an electrolyte solution that has different corrosion potentials. The very low concentrations of Cr and Ni ions (ppm level) in the immersion test results of the Saline solution after various immersion intervals are depicted in Figure 7. For eight weeks, Cr was detected in every medium tested at concentrations up to 0.058 ppm. Ni was also detected in all media, with the highest concentration reaching 0.199 ppm over a six-week period. Whereas in the application of implants that are biodegradable, according to Nasution et al. and Zhang et al. [20] [35], The amount of Fe ions released is very important to maintain the level of safety in the human body. The highest average concentration of Fe ions in the eighth week was 17423 ppm, and this is still considered safe for the human body, according to Zhu et al. [34].

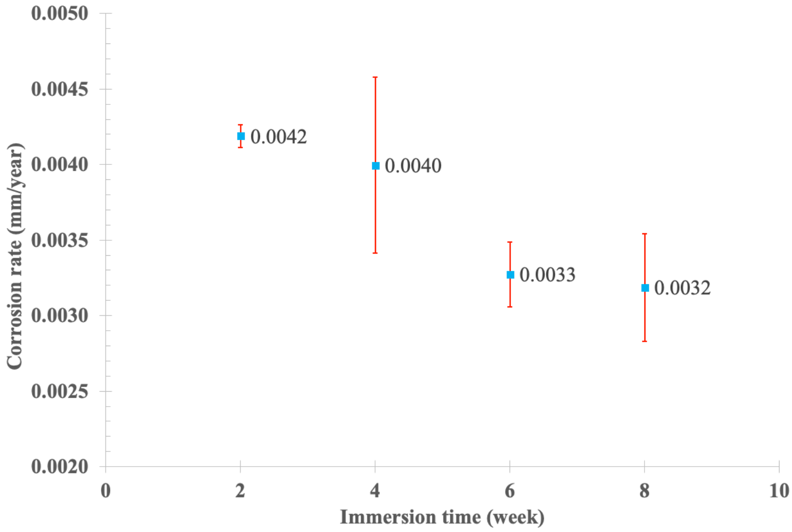


Figure 4. Corrosion rate during immersion test.

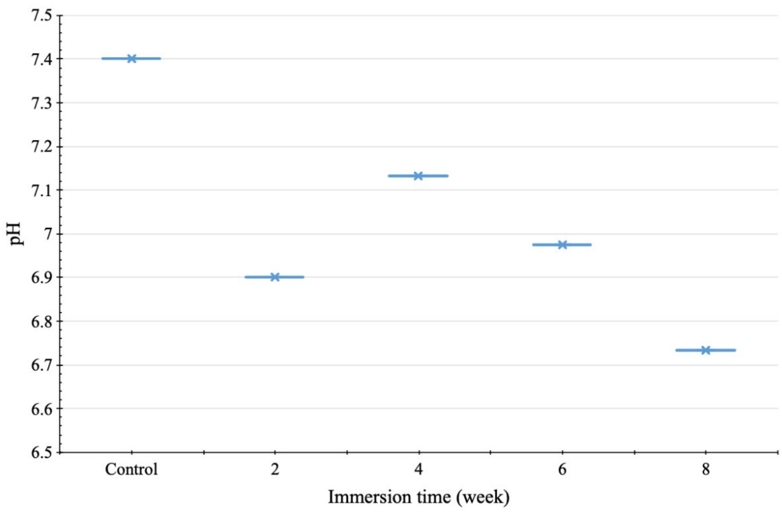


Figure 5. pH changes of Saline media of the tested samples after 2, 4, 6, and 8 weeks of immersions.

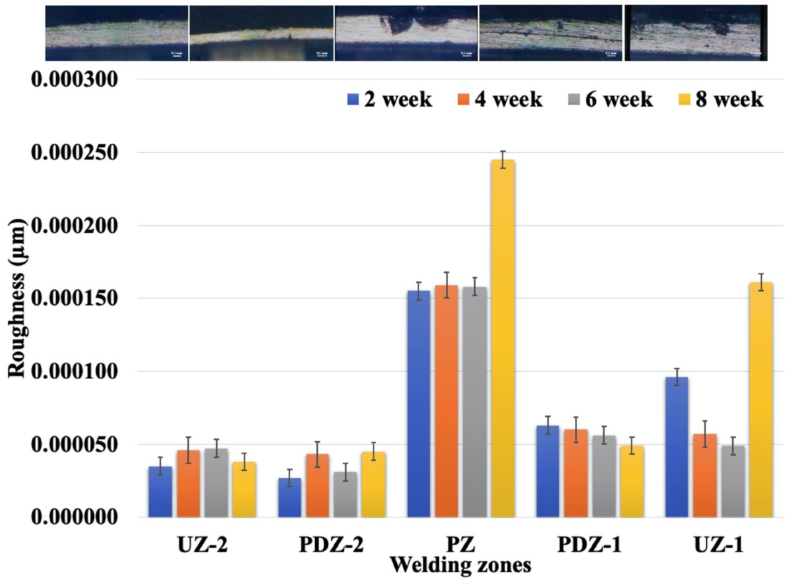


Figure 6. Corrosion morphologies and surface roughness values of the welding zones of the samples after immersion for eight weeks.

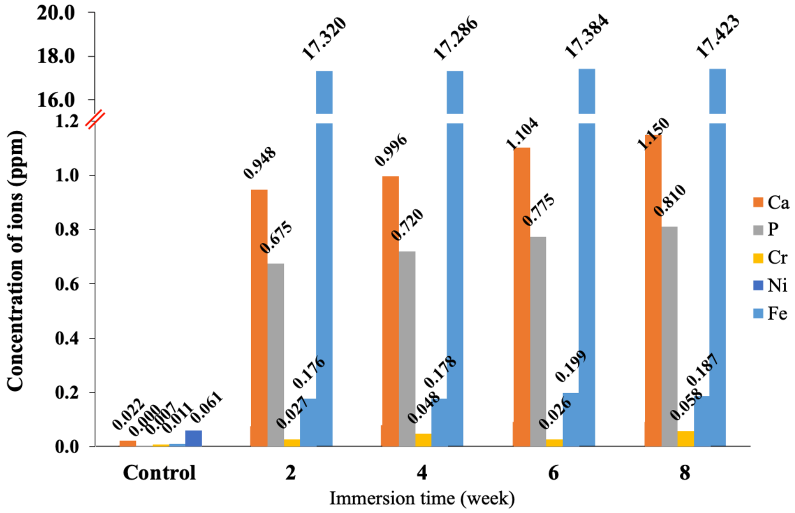


Figure 7. The concentration of ions released into the Saline solution.

Cytotoxicity

Figure 8 shows the viability of HSF cells after the incubation at 1, 3, and 5 days. In general, all specimen zones demonstrated good tolerance with HSF greater than 75% cell viability. Similar observations were found by Nasution et al. on the viability of human osteoblast cells (NHOst) against pure Fe, where all zones produce more than 75% cell viability [5]. Besides, these results are in line with a study on Fe-based material with smooth muscle and endothelial cells with no record of cell inhibition growth [36]. Huang also mentioned that Fe ions had almost no effect on the metabolism of the human umbilical vein [25]. Therefore, the welded metals of SS202 and low carbon steel were concluded to be non-cytotoxic, thus suitable to be adopted as implant materials.

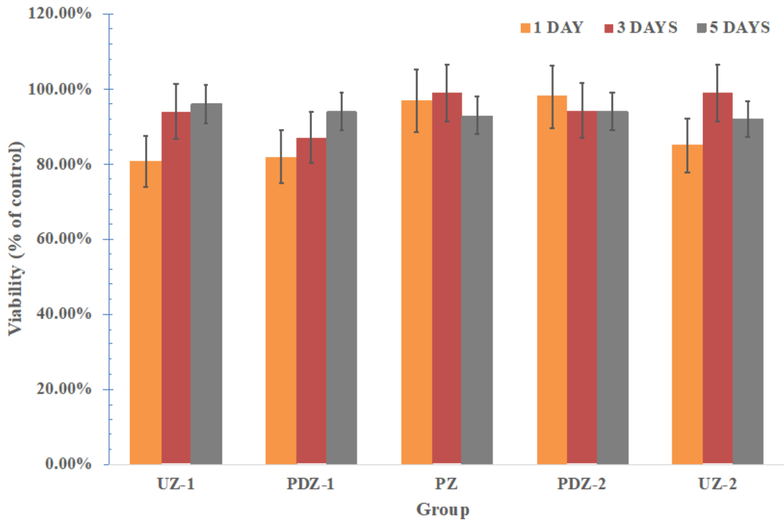


Figure 8. Viability of HSF cells for all samples in each zone.

CONCLUSION

The friction welding machine with continuous drive with small welding parameters was successfully made to specifications for 4000 rpm rotation, test sample diameters up to 12.7 mm, and hydraulic pressures up to 24.5 MPa. In this study, carbon steel and stainless steel (SS 202) were successfully welded with welding parameters at a rotating speed of 4000 (rpm), friction pressure 0.98 (MPa), forging pressure 4.90 (MPa), and friction time 8 (s). This parameter produces a maximum tensile strength of 874.46 MPa and yield strength of 800.67 MPa, and this welding parameter also produces a heat input of 0.3 J/s. The results of the bending test were broken at the welding interface (WI). An initial cytocompatibility evaluation on HSF confirms that all zones are compatible and non-toxic to these cells.

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