Investigation of loosening resistance under assembly conditions using different locking chemicals and lock nuts

Baybars Sarıca¹, Tolga Aydın¹, Samed Enser^{*1} and Umut Ince¹

¹Norm Fasteners, R&D Center, AOSB, Cigli, İzmir, Türkiye

Article Info	Abstract
Article history: Received 22.12.2023 Revised: 18.04.2024 Accepted: 03.05.2024 Published Online: 14.06.2024 Keywords: Bolt Lock Nut Locking Chemical Assembly Junker vibration test	Fasteners are subjected to repetitive and variable loads in the areas where they are used. This situation creates some risks such as loosening or fracture of fasteners, result the failure of the joint. Locking chemicals applied on the bolt to prevent loosening that may occur under assembly conditions are used extensively, especially in the automotive industry. By using locking chemicals selected in accordance with the assembly area and fastener, it is aimed to prevent the loosening of the assembly parts under vibration. There are commercial products that offer different properties that can be used in the mass production of locking chemicals. Within the scope of the study, the performance of three different commercial locking chemicals against loosening was investigated and used standard M8 bolts and nuts. In addition, the M8 DIN 980 lock nut, which is used in joints that are at risk of loosening in vibrating areas, was also added to the studies. Due to the effect of the coefficient of friction on the clamping load, all fasteners were coated with the same coating to ensure equivalence in installation conditions. The vibration performances of the locking chemicals were investigated with the Junker vibration test. The results were analyzed comparatively and the joint condition with the highest vibration resistance was determined. As a result, it was observed that the use of locking chemicals and lock nuts increased the loosening resistance.
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1. Introduction

Bolt-nut connections are one of the oldest and most frequently used joint methods. Bolt-nut connections, which are preferred in many sectors including automotive and white goods, are exposed to loads and displacements at high cycle numbers and for long periods. If the appropriate clamping load is not created during the tightening process, loosening may occur at low cycle numbers. Even if there is no problem in the tightening process, it is exposed to dynamic and static loads according to the conditions of the application area. As a result of the radial contraction of a bolt subjected to dynamic loads and radial expansion of the nut with the amount of Poisson's ratio deformation, the bolt produces a radial micro-slip at both the bolt-nut thread interface and the bearing surface during axial stress variation and the loosening problem occurs over time (Figure 1) [1].

Especially in the critical design points, loosening of the joint must not occur. The introduction of robotic systems in mass production factories in Industry 4.0 applications, is aimed to minimize the errors that may occur in the assembly area. The performance of the joint is affected by the bolt-nut parts as well as the assembly conditions. The yield point, friction coefficient, clamping length and moment of inertia of the fasteners are the main parameters of the fractures/loosening that may occur in the connection. In the assembly area, proper tightening in accordance with the bolt-nut quality class is of great importance. In a study conducted in 2010, it was stated that the coefficient of friction changes due to wear during repeated tightening of the fastener and therefore, when tightened at a certain torque value, the clamping load realized in the assembly decreases. Furthermore, if an axial load is also acting on a joint that is experiencing radial slippage, it has been reported that lock nut can continue to self-loosen, leading to loosening [2].



Figure 1. Sliding zones in nut-bolt threads under vibration

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Corresponding Author: Samed Enser E-mail: samedenser@normfasteners.com

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In another study conducted by NASA, vibration tests were carried out by simulating the rocket conditions of a bolt-nut connection mechanism with a safety wire that would not lose its preload, considering the vibrations generated in a rocket. As a result of the study, it was stated that radial movement caused by vibration and structural forces was the main cause of loosening [3]. There are also studies in the literature on modeling the selfloosening behavior and thus predicting the loosening behavior of bolted joints without a test. In a 2021 study, the self-loosening behavior of bolted joints was investigated by numerically and experimentally. The numerical analysis showed that the existing analytical model was unable to predict the experimental loosening behavior therefore the analytical model was modified with the help of a finite element model. In this way, the prediction of self-loosening behavior was increased from 58.3% to 73% [4]. In a study at Toyota Automotive Co., the selfloosening behavior of bolt-nut connections subjected to tensile load was investigated and the loosening behavior was described with the fundamental material strength equations. As a result of the study, it was suggested that the clamping load on the joint is removed as instant due to the tensile force, and in the absence of this load, the bolt rotates counterclockwise due to the torque generated by the axial stress and loosening occurs. However, it is said that this type of loosening rarely occurs in steel bolt-nut connections [5]. In another study in 2022, a radial anti-loosening precision locked-nut was used and tested under three different target axial force conditions. With this test, the stiffness of the assembly was measured. At the end of the study, the authors concluded that the use of precision locked-nuts in fastener systems has improved assembly properties by providing stable design stiffness under dynamic vibration. The authors tested parameters such as tightening-loosening torque, axial force ratio and anti-loosening ratio of the nut in different vibration conditions and showed that the precision locked-nut improves the dynamic stability of the system [6]. In a 2021 review article, systems and methods to prevent loosening are listed. While this study was carried out, methods were categorized under two headings; traditional methods and newly developed methods. While, the authors have categorized the traditional methods evaluated and compared as anti-loosening structures in the form of a washer and nut, the new-style anti-loosening structures methods under three different headings. These are; changing the thread of the nut or bolt, changing the bearing surface of the nut or bolt head, and changing the material type of the nut or bolt. At the end of the study, it was mentioned that the methods have advantages/disadvantages against each other and it was suggested that there is no single method that can solve all loosening problems yet. The authors did not mention the use of locking chemicals in the anti-loosening method [7]. In 2021, another paper investigated how to improve both the antiloosening and fatigue strength of a bolt-nut connection at low cost. M12 pitch 1.75mm bolts and nuts were used in the study. Different nut lengths and standard pitch length of 1750 µm were given different length (α) deviations and the fatigue and loosening performance of the connection was investigated. At the end of the study, the authors reported that the joints with longer nuts and smaller pitch deviations had both improved fatigue performance and increased loosening resistance [8]. A study in 2007 investigated the self-loosening performance of Teflon and adhesive coatings. Two different adhesive coatings and two different Teflon coatings were studied and compared with uncoated scenarios. The main objective of the study was to find a relationship between the self-loosening resistance of the

tested scenarios and the friction coefficients. Teflon coatings were found to reduce the coefficient of friction, but not the loosening resistance of the joint. Adhesive coatings were said by the authors to increase the loosening resistance of the assembly. However, it was reported that the adhesive coatings used lost their properties over time under transverse loading [9].

Vibration loads are the major factor determining the loosening behavior of a bolt. Under certain conditions, tightened bolts are observed to have a loosening problem. The sliding behavior of the bolt-nut connection and the transverse rotation of the nut under transverse load is shown in Figure 2. When an external shear force acts on the joint, time-varying forces in the form of tension and compression act on the parts tightened between the bolt head and the nut. The external load is divided into three components due to the inclination angle of the threads and the flange angle. The first of these components along the axis of the bolt, the other in the radial direction and the last one acts tangentially to the surfaces. The force component acting along the bolt axis extends and deforms the shaft, while the radial force bends the thread profile. The radial force creates a moment in the opposite direction to prevent warping from occurring [10, 11].



Figure 2. Sliding behavior of bolted joint under vibration [10]

In addition to the appropriate bolt-nut joint, locking chemicals are also used to prevent loosening caused by both bolt-nut and assembly area conditions in the joint areas and to increase the operating performance of the fittings in cases where sealing is desired. Figure 3 shows the method of application of a commercial locking chemical. Each commercial locking chemical has its own specific application which methods are specified by the manufacturer [9, 12].



Figure 3. Locking chemical distribution on the bolt [13]

As can be seen, there are many studies on resistance to loosening in the literature. However, there is no comparative study that reveals the performance differences between newly locking chemicals. Moreover, it is also unknown how the locking chemicals perform compared to mechanical locking methods. In this study, bolts, standard nuts and lock nuts with the same coating and constant coefficients of friction were used. In order to investigate the effect of locking chemicals on vibration resistance, three different commercial locking chemicals were applied to the bolts and vibration tests were performed. After clamped load was applied with a digital wrench, the bolts were subjected to vibration in the transverse direction for a certain displacement and cycle duration with Junker tester. During the Junker vibration test, cycle-based changes in the clamping load value were recorded so that the loosening rate data of the assembly combination were obtained and compared with each other. The combination giving the highest vibration resistance among the two different nuts and three different locking chemical applications used in the study was determined after the comparative results.

2. Materials and methods

In order to compare the performance of locking chemicals, the loosening resistance of bolt-nut connections was evaluated by the Junker vibration test. The bolts used for the test are M8x1,25 8.8 DIN 933 standard bolts produced by Norm Fasteners. M8x1,25 8.8 DIN 934 standard hex nuts and DIN 980 lock nuts tightened hex nuts produced within Norm Fasteners were also used for fastening. The bolts and nuts used in the tests were coated with a zinc-plated coating by dip-spin method and then top-coated to ensure that their surface quality and roughness were the same. In this way, a constant coefficient of friction in the range of 0.09-0.12 was obtained in the bolts and nuts. The clamping length of the bolts was kept constant at 53 mm. This ensured that the internal threads of the nuts were in full contact with the applied chemicals under assembly conditions. The test procedure to be followed throughout the study is shown in Table 1.

Table 1. Test Procedure.						
Locking Chemicals	Bolt	Nut	Test Condition			
Lock. Chem. 1	DIN 933	DIN 934 / DIN 980	5 Hz, 53 mm clamp length			
Lock. Chem. 2	DIN 933	DIN 934 / DIN 980	2000 cycle			
Lock. Chem. 3	DIN 933	DIN 934 / DIN 980	0,7 mm displacement			

Technical specifications of the locking chemicals used are shown in Table 2.

 Table 2. Technical Specification of Locking Chemicals.

Locking Chemicals	Active Substance	Friction Coefficient	Micro- Capsule Content
Lock. Chem. 1	Toluen	0,16	+
Lock. Chem. 2	Acrylic	0,24	+
Lock. Chem. 3	Polyamide	0,3	

Although the coefficient of friction of the bolted joint is constant through the coating of the bolts and nuts, the joints tightened to the same torque value do not reach the same clamping load due to the friction values of the locking chemicals being compared and the prevailing torque values of the lock nuts. Since the clamping load is the main factor observed in the loosening resistance, the bolted joint was tightened to reach a constant clamping load for comparison in all scenarios. A digital wrench was used as a tightening tool. When calculating the constant clamping load, the minimum breaking torque value of 33 Nm specified in the ISO 898-7 standard was taken as a reference [14]. The maximum safe clamping load $10^{+0.5}_{-0}$ kN was tightened for the scenario with the highest coefficient of friction (μ =0.3). When calculating this value, the Equation 1 from ISO 16047 "Fasteners - Torque/clamp force testing" standard was used [15];

$$\mu = \frac{T/F - P/2\pi}{0.577d_2 + 0.5D_b} \tag{1}$$

Where T, F, P, d2 and Db are torque (Nm), clamping load (N), pitch (mm), basic pitch diameter of the thread and effective bearing surface (mm) respectively.

Each locking chemical used in the tests was applied in the method and range recommended by its manufacturer. Bolts with three different locking chemicals are shown in Figure 4. Locking Chemical 1, fastener adhesive, are microencapsulated, room temperature curing locking chemicals that improve the loosening resistance of threaded fasteners. The adhesives are designed to be poured onto fasteners and dried. Locking Chemical 1 contains a microencapsulated epoxy resin suspended in form. When the fastener is tightened, the microscopic capsules of the epoxy resin are reacted by the force between the threads and the reactant components initiate a chemical reaction that locks the parts together. An image of Locking Chemical 1 applied to the bolts used in the project is given in Figure 4 (a). Locking Chemical 2, like Locking Chemical 1, contains epoxy resin suspended in its own form. The microcapsules are released by compression and shear stress during assembly and create a coating on the bolt threads (Figure 4 (b)). Locking Chemical 3, like Locking Chemicals 1 and 2, is applied on the bolt threads to create resistance to vibration in the clamping area. The radial tension created by the elastic deformation of the product causes the locking chemical 3 to maintain the clamping position through increased friction. Locking Chemical 3 has some differences compared to Locking Chemicals 1 and 2. Locking Chemicals 1 and 2 have an adhesive effect where the epoxy granules in their structure spread over the bolt threads after torque is applied into the clamping area. The dispersed epoxy granules need to be cured at room temperature in the assembly area. Locking Chemical 3 is a powder sprayed on bolt and nut threads and can be used in the assembly area without the need for curing. This is because Locking Chemical 3 does not have epoxy granules in its structure like the other chemicals, but it is a locking chemical that forms an elastic, wear-resistant blue polyamide coating on the threads by spraying (Figure 4 (c)).

In this study conducted at Norm Fasteners R&D Center, tests were carried out in the Junker test machine with $10^{+0.5}_{-0}$ kN preload, 500 rpm speed and 2000 cycle test parameters. At least 5 tests were performed for each group. The test setup illustration and test machine of the Junker tester are shown in Figures 5 (a) and (b) respectively [16].



Figure 4. Bolts with Locking Chemicals (a) 1, (b) 2 and (c) 3



(a)



Figure 5. (a) Test set-up illustration and (b) Junker tester

3. Results and discussion

In the first step of the study, vibration tests were performed using DIN 933 (standard hex bolt) bolts and DIN 934 (standard nut) and DIN 980 (lock nut) nuts without locking chemical for reference. The average test results for the tests without locking chemicals are shown in Figure 6. Here the results are shown as a graph of clamping force versus cycle. It was observed that after the operation with the standard nut, the initial clamping load decreased from 10 kN to 7.2 kN due to loosening in the transverse direction. When the results obtained after the vibration test with the lock nut are compared with the test with the standard nut, it is seen that the reduction in the clamping loads is quite limited and there is no complete relaxation in 2000 cycles. Unlike standard nuts, while tightening, lock nuts are assembled encountering a mechanical resistance on the bolt threads which is prevailing torque. The additional mechanical resistance creates a structure that is resistant to loosening. Compared to standard nuts, the positive effect of the mechanical resistance obtained with lock nuts on the loosening resistance can be seen.



Figure 6. Average results of locking chemical-free vibration test with two different nuts.



Figure 7. Average results of vibration test with Locking Chemical 1



Figure 8. Average results of vibration test with Locking Chemical 2

After the reference test which has no locking chemical, Locking Chemical 1 was tested first. After the microcapsules in the epoxy structure were tightened and dispersed on the bolt, they were left for 1 day as they needed to cure. The average test results for the tests where Locking Chemical 1 was applied are shown in Figure 7. When the results with standard nut and Locking Chemical 1 are examined, it is seen that the clamping load decreased up to 9.4 kN but never went below this value. If we look at the result of the lock nut, it is seen that the clamping load decreased up to 9.8 kN values but never went below this value. There was also some difference between the average initial clamping load levels in the tests. The results of the studies using Locking Chemical 2 are shown in Figure 8. Locking Chemical 2, like Locking Chemical 1, is a chemical that requires curing to achieve the desired mechanical properties. After the test with the standard nut and Locking Chemical 2, it was seen that the clamping load loosened up to 9.5 kN, but on average, the clamping load remained constant after 1000 cycles. When the vibration test with the lock nut and Locking Chemical 2 is examined, it is seen that it does not go below the clamping load of 9.6 kN, similar to the results of the standard nut.



Figure 9. Average results of vibration test with Locking Chemical 3



Figure 10. Vibration test results of (a) standard and (b) lock nuts with 3 different locking chemicals

After the applications of Locking Chemicals 1 and 2, which were cured and ready for use, the studies continued with Locking Chemical 3, which is a locking chemical that is ready for use directly after tightening. The test results with Locking Chemical 3 are given in Figure 9. It was observed that the clamping load value in the Locking Chemical 3 application with the standard nut showed different results after 5 tests. In four tests, it was observed that it oscillated with a relatively large amplitude between 9.5 - 8.8 kN, while in one test, the clamping load value decreased to 6.0 kN. Considering the studies conducted with the standard nut, it was clearly observed that it exhibited more inconsistent loosening performance than the cured Locking Chemicals 1 and 2 applications. When the results of the Locking Chemical 3 application with the lock nut were examined, it was observed that unlike the test results with the standard nut, the combination of Locking Chemical 3 and the lock nut showed both consistent results and the clamping load remained above 9.6 kN throughout the cycles.

Table 3. Result of locking of	chemicals	and lock	nut
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performances				
Locking Chemicals	Nut	Loosening Ratio %		
No Chem. (ref)	934	29		
No Chem. (ref)	980	2		
Lock. Chem. 1	934	6		
Lock. Chem. 2	934	5		
Lock. Chem. 3	934	12		
Lock. Chem. 1	980	2		
Lock. Chem. 2	980	4		
Lock. Chem. 3	980	4		

4. Conclusions

Within the scope of the project, the resistance to loosening of three different locking chemicals, which are frequently used with bolted joints in automotive, was investigated. The test results of standard nuts and lock nuts are shown in Figures 10 (a) and (b) together with all locking chemicals for comparison. In addition, the performances of the locking chemicals are tabulated in Table 3. When the results were analyzed;

- When standard nut without locking chemicals is used, the clamping loads decreased approximately 30% after 2000 cycles in vibration tests. Whereas the clamp load decreased by only 2% when the lock nut was used.
- When locking chemicals were used, the maximum loosening rate was approximately 5% for Locking Chemicals 1 and 2, which contain microcapsules, provide locking with adhesive properties and therefore require curing time. On the other hand, a 12% loss in the clamping load was realized with Locking Chemical 3, which provides only a mechanical locking to improve the loosening resistance.
- Considering these results, locking chemicals applied to standard nuts have been shown to successfully improve loosening resistance.
- When the tests with lock nut and locking chemicals together were examined, it was observed that the effect of locking chemical applications on the loosening resistance did not provide a significant difference in the test conditions compared to the standard nut applications. Although it was expected that the using of a lock nut and locking chemicals together would have a positive effect on the loosening resistance, it was clearly seen that it gave similar results to the scenarios with only used one of the solutions.

In short, it was observed that the use of only the lock nut provided as much loosening resistance as the standard nut and locking chemical application. Interpretation of the performance of locking chemicals could only be done for the installation scenarios with standard nuts where significant differences could be observed.

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Author contributions

All authors reviewed and approved final version of the manuscript. Baybars Sarıca: Investigation, Validation, Data curation, Formal analysis. Tolga Aydın: Conceptualization, Visualization, Formal analysis. Samed Enser: Writing – editing & original draft, Resources, Supervision. Umut İnce: Investigation, Resources, Methodology, Supervision.

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