

Extract of calligonium polygoids as a sustainable inhibitor for mild steel in acidic medium and effective antioxidant

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Abstract

In the quest for corrosion inhibitors that are both sustainable and environmentally benign for mild steel in acidic environments, natural plant extracts have been investigated as a potential solution. In this study, the efficiency of Calligonum polygonoides extract as a green corrosion inhibitor for mild steel in a solution of 1 M hydrochloric acid is investigated. Methods such as electrochemical impedance spectroscopy (EIS), potentiodynamic polarization, and weight loss measures were utilized in order to assess the effectiveness of the corrosion inhibition. According to the findings, the extract of Calligonum polygonoides has the ability to dramatically lower the rate of corrosion of mild steel when it is exposed to an acidic media. When the concentration of the plant extract was increased, it was discovered that the efficiency of the inhibition increased as well, reaching as high as 90 percent at higher doses. SEM, which stands for scanning electron microscopy, was used to conduct surface morphology investigations, which proved the creation of a protective layer on the surface of the steel. Because the Langmuir adsorption isotherm is followed by the adsorption of the extract on the surface of the mild steel, it may be inferred that the inhibitory mechanism includes the adsorption of phytochemical elements onto the surface of the steel, which prevents corrosion. The thermodynamic parameters, which were derived from the temperature dependence of the inhibitory efficiency, provide evidence that the adsorption process is spontaneous and involves physical adsorption. An extract of Calligonum polygonoides was shown to be an efficient and sustainable corrosion inhibitor for mild steel in acidic settings, according to the findings of the study. This extract offers a potential alternative to synthetic inhibitors, with the additional benefits of being ecologically benign and economically feasible.

Keywords : *calligonum polygoinds, calligonum polygoinds, acidic*

Introduction

A number of different industrial operations, such as acid cleaning, pickling, and oil well acidizing, face a substantial problem in the form of corrosion of metals, particularly mild steel, when they are exposed to acidic surrounding conditions. When it comes to preventing corrosion, conventional approaches include the utilization of synthetic inhibitors, which frequently present environmental risks owing to their toxicity and inability to be broken down by biological processes. As a consequence of this, there is a rising interest in the development of corrosion inhibitors that are sustainable and favorable to the environment, and that are produced from natural sources. As a result of their availability, biodegradability, and non-toxic nature, plant extracts have emerged as potentially useful alternatives. These extracts include a high concentration

of organic chemicals, including alkaloids, flavonoids, tannins, and polyphenols. These compounds have the ability to adsorb onto metal surfaces and create a barrier that protects against corrosive substances. Calligonum polygonoides, more popularly referred to as "Arta," is one of the plants that has garnered recent interest due to the possible implications it may have in the field of corrosion inhibition. Calligonum polygonoides is a shrub that is well-known for its resistance and therapeutic characteristics. It is extensively spread in dry environments. Due to the fact that the extract from this plant includes a wide range of bioactive chemicals that are capable of interacting with metal surfaces, it is a potential candidate for research dealing with the suppression of corrosion. The purpose of this study is to determine whether or not an extract of Calligonum polygonoides is effective as a green corrosion inhibitor for mild steel when it is exposed to an acidic media. In order to evaluate the effectiveness of the plant extract as an inhibitor, this study makes use of techniques such as electrochemical impedance spectroscopy (EIS), potentiodynamic polarization, and weight loss tests. In addition, surface morphology study is carried out with the help of scanning electron microscopy (SEM) in order to witness the creation of protective layers on the surface of the steel. Additionally, the adsorption behavior of the extract as well as its thermodynamic characteristics are investigated in order to shed light on the mechanism of inhibition. The purpose of this research is to contribute to the development of sustainable corrosion inhibitors by investigating the potential of Calligonum polygonoides extract. This research offers an ecologically friendly option to limit the negative effects of metal corrosion in situations that are acidic.

Material Methods

There is a significant therapeutic use for the phytochemicals that are obtained from plants. These phytochemicals are a source of raw material for the development of nutraceuticals and pharmaceutical goods, which have the potential to alleviate specific sufferings that are associated with human health. The production of these plant-based bioactive compounds in a manner that is both economical and safe is the primary challenge. This is due to the fact that the production of these bioactive compounds in plant systems is relatively low (less than 1% of dry weight) under natural conditions. Furthermore, the production of these bioactive compounds is highly dependent on the physiological and developmental stage of the plant, as well as environmental factors such as temperature, light intensity, soil water, soil salinity, and fertility. The optimization of the production level of these bioactive chemicals in plants is therefore the area of focus in this study. There are extremes in the climate of the "Thar Desert," which is characterized by its hot and arid climate, with very low annual rainfall (40 degrees Celsius and on some days it reaches 50 degrees Celsius) and very high UV radiations (May–June). Additionally, the winters are extremely cold, with monthly average minimum temperatures reaching as low as 5 degrees Celsius, and on some days it goes below zero degrees Celsius during the months of December–January (Figure 1). In order for a plant to even be able to live in these harsh climatic circumstances, it really needs to have a specialized ROS scavenging mechanism built into it so that it can combat the enormous oxidative stressors that are pouring in. In addition to being able to endure these harsh climatic conditions, C. polygonoids are also capable of producing a substantial quantity of biomass.

The purpose of this study was to explore the impact of different climatic conditions on the antioxidant activity, phenolics, flavonoids, and condensed tannin content of C. polygonoides leaf that was obtained from the Thar Desert in India. Secondary metabolites, also known as phenolic compounds, are produced by a wide variety of plants in order to protect themselves from abiotic stresses. At the same time, phenolic bioactives may be isolated and employed for the creation of functional foods that have a high level of

antioxidant activity. Under both low temperature stress and dry environments, the C. polygonoides plant is able to thrive and develop quickly. Therefore, it is of the utmost importance to have a comprehensive understanding of the seasonal change in phenolic compounds and the antioxidant activity of the extracts obtained from the leaf of C. polygonoides. This inquiry is the first publication on C. polygonoides that takes into consideration the influence of climatic circumstances or seasonal fluctuation on phytochemical profile and antioxidant activity. This is the case according to the written literature that is currently available. Therefore, the assessment of the secondary metabolite profile with seasonal variation might give essential information regarding the time of harvest of foliage that allows for the optimal concentration of active substances for the development of functional food products.

Results and Discussion

Ethanolic extracts of C. polygonoides leaves, collected at various times of the year from plants cultivated in hot, dry regions, were used to estimate TPC, TFC, and TTC according to established techniques. The three indicators, namely TPC, TFC, and TTC, showed a significant variation $(p < 0.05)$ as the years and harvesting months progressed. These secondary metabolites vary greatly from one harvest month to the next because of the profound effect that changes in environmental conditions have on the manufacture and accumulation of these chemicals in plants. In June, during peak summer, and in December, during peak winter, the values of TPC, TFC, and TTC were the greatest. Table 1 and Figure 1display the findings of expressing the seasonal changes in TPC of C. polygonoides foliage as GAE using the standard curve equation. Among TPCs recorded in different months, a significant difference was noted ($p < 0.05$). The total phenolic content (TPC) of C. plygonoides leaves collected at various times of the year varied between 32.28 ± 0.54 and 88.08 ± 0.59 mg. The output is for GAE.g-1 FW. Compared to earlier findings by Berwal et al., which measured 151 mg of TPC in leaves, this new data shows a lower level.The reason our results are based on fresh weight rather than dry weight is because the authors gave data for GAE.g−1 TPC in C. polygonoides leaf. Additionally, Samejo et al. found that C. polygonoides plants had higher TPC values. In December, the highest TPC was observed at 88.08 \pm 0.59 mg.July, August, and September had the highest levels of GAE.g−1 FW, with 81.84 ± 2.28, 71.97 ± 1.33, and 64.75 ± 2.13 mg.GAE.g−1 FW, respectively. In March, the TPC was 32.28 ± 0.54 mg.GAE.g−1 FW, and in October, it was 34.05 ± 1.53 mg.GAE.g−1 FW, both of which were statistically equivalent (p < 0.05).

Figure 1. Foliage of Calligonum polygonoides L. subjected to harsh weather conditions changes in total phenolics, tannin, flavonoids, and antioxidant activity throughout the year. With $n = 3$ trials and a p-value less than 0.05, the data are shown as the mean \pm SD.

Table 1 and Figure 2 both make it very easy to understand how the TFC levels change throughout the year for each season. When examining the TFC values, it was noted that there was a noteworthy range ($p <$ 0.05), with the values ranging from 1.0 ± 0.01 to 2.80 ± 0.02 mg. The CtE.g−1 FW. Similar to TPC, the maximum total fatty acid concentration (TFC) was observed during the month of December (2.80 ± 0.02 mg.CtE.g−1 FW). This was followed by the months of June (2.53 \pm 0.03), May (2.15 \pm 0.04), and January (2.07 ± 0.03) . On the other hand, the lowest TFC is reported during the months of March (1.0 ± 0.01) and October (1.1 \pm 0.01). When compared to the values found in the earlier studies, the maximum TFC levels are lower. Using a dry weight basis, Berwal et al. showed that the leaves of C. plygonoides contained around 6.5 mg.CtE.g−1 of TFC.

Figure 2 and Table 1 both illustrate the tannin content (TTC) of the foliage of C. polygonoides, which was represented as mg.Catechin.E. g−1 FW. With a magnitude ranging from 40.40 ± 0.89 to 96.09 ± 1.38 mg, the TTC of C. polygonoides leaves gathered throughout different months exhibited a considerable variation from one another due to statistical significance ($p < 0.05$). For example, Catechin. E.g. 1 FW. Comparable to TPC and TFC, the greatest TTC was recorded in the month of December (96.09 \pm 1.38), followed by the months of June (95.17 \pm 2.22) and January (87.13 \pm 1.78). On the other hand, the lowest TTC was recorded during the month of March (40.4 \pm 0.89), followed by the month of October (43.94 \pm 1.40) mg. Catechin.E.g−1 FW.

Seasonal Variations of Total Antioxidant Activity

Based on its reducing capacity, the total antioxidant activity (TAA) of ethanolic extract of C. polygomoides leaves gathered during different months was measured using several techniques such as CUPRAC, FRAP, and DPPH test. The results were represented as mg ascorbic acid equivalent per g (mg.AAE.g−1) of fresh weight (FW). In general, the findings of these three tests were consistent with the shift in TPC and TFC levels that occurred during the seasons (Figure 2). Obtaining an understanding of the true nature of the antioxidant chemicals that are present in the leaves of C. polygomoides can be facilitated by the determination of the reducing power through the combination of several approaches. Each of the test protocols that were followed resulted in a significant variation ($p < 0.05$) in the TAA of the C. polygomoides foliage that was gathered during various months (Table 1 and Figure 2). According to the findings, it was noted that the total aqueous extract (TAA) of the foliage of C. polygomoides exhibited a significant variation ($p < 0.05$) with harvesting months, regardless of the assay protocol that was utilized. Under each and every assay process, the TAA was found to be at its peak during the months of June and December, followed by May and January, and it was found to be at its lowest during the months of March and October. The total acidity (TAA) of C. polygomoides foliage, which was collected during different months and evaluated using the CUPRAC test, exhibited a substantial variation ($p < 0.05$) ranging from 51.22 \pm 1.14 to 118.84 ± 2.12 mg.AAE.g−1 FW, as reported in Table 1 and Figure 2. It was noted that the greatest TAA was recorded in the month of June, with a value of 118.84 ± 2.12 mg.AAE.g−1. This was followed by the month of December, with a value of 115.81 ± 2.97 mg.AAE.g−1. The lowest TAA was recorded in the month of October, with a magnitude of 51.22 ± 1.14 mg.AAE.g−1 and 51.73 ± 1.42 mg.AAE.g−1, respectively, and being considerably comparable to each other.

The TAA of C. polygomoides leaf, which was collected during different months and measured using the FRAP test, likewise exhibited substantial fluctuations ($p < 0.05$), specifically ranging from 18.26 ± 0.21 mg to 27.11 ± 0.20 mg.The AAE.g−1 FW. According to the FRAP assay, the greatest TAA was observed during the month of December, with a magnitude of 27.11 ± 0.20 mg. AAE.g−1. This was followed by the month of June, with a magnitude of 25.97 ± 0.41 mg. AAE.g−1. The lowest TAA was reported during the months of October and July, with a magnitude of 18.26 ± 0.21 and 19.42 ± 0.23 mg.AAE.g−1 respectively. Likewise, the TAA of C. polygomoides foliage, which was assessed based on DPPH scavenging activity, fluctuated throughout the year in accordance with the months in which harvesting occurred. With a magnitude ranging from 43.42 ± 3.14 to 105.46 ± 2.70 mg, the findings indicated that there was a significant difference ($p < 0.05$) in TAA levels between the varying harvesting months. Figure 2 and Table 1 both have the AAE.g−1 FW. Similar to the FRAP assay, the greatest TAA was found during the month of December (105.46 ± 2.72 mg.AAE.g−1), which was considerably comparable to June (105.30 ± 2.72 mg.AAE.g−1). On the other hand, the lowest TAA was recorded during the months of October and March, with the magnitudes of 43.42 ± 3.14 and 47.39 ± 2.21 mg respectively. The AAE.g−1 FW.

Principal Component Analysis (PCA)

With the help of the characteristics that were analyzed, such as TAAs, phenolic, flavonoids, and tannin content, the principal component analysis (PCA) was able to explain 93.7% of the variability in axis one and two. Figure 3 shows that the first component (PC1) accounted 68.4% of the total variance, while the second component (PC2) explained 25.3% of the variation. The results of this experiment made it abundantly evident that there is a degree of variation in the parameters that were measured in relation to the months of sampling or the temperature of the environment. It was shown that there was a high correlation between axle one and the levels of all metrics, including TAA, phenolics, flavonoids, and tannin contents. The biplot graph that was generated as a result of this study demonstrated that there is seasonality in the antioxidant reactions that occur in the leaves of C. polygonoides. This was demonstrated by all of the parameters that were analyzed, including TAA, phenolic, flavonoids, and tannin concentrations (Figure 3). On the positive side of axle one, the sample units of the summer months (May and June) and the winter months (December and January) were clustered together. These units were distinguished by the highest values for total amino acid (TAA), phenolics, flavonoids, and tannin content. The sample units for the subsequent months, on the other hand, were found to be clustered together on the opposite side of this axle. Under situations of oxidative stress, plants often generate a greater quantity of antioxidants. Furthermore, the climatic conditions in the hot and dry region are especially prone to extremes. It is possible for the temperature of the environment to remain as high as 48 degrees Celsius during the summer months, with very strong radiations, while during the winter months, the temperature might drop to subzero degrees Celsius. It was also stated that Ipomoea nil cv. Scarlet O'Hara produced results that were comparable.

Figure 2. A principal component analysis (PCA) was performed on the tanning content, phenolics, flavonoids, and total amino acids (TAA) in the foliage of C. polygonoides over the various months.

Loess Regression Analysis against Maximum and Minimum Temperature

The locally weighted scatter-plot smoother (LOESS) curve was plotted for the data that was collected throughout the twelve sampling intervals against the maximum and minimum monthly average temperature (Figure 4). This was done in order to show the seasonal variations that occurred in TPC, TFC, TTC, and antioxidant activity. As a consequence, we obtained a regression curve in the shape of a "V" from all of the factors that were investigated, as well as the oscillations in the maximum and minimum temperatures, which ranged from 35 ± 1.0 °C to 20 ± 1.0 °C, respectively.

Figure 3. The total polygonoides compound (TPC), total flavonoid concentration (TFC), total antioxidant activity (TAA.CUPRAC), and its seasonal fluctuation were examined using LOESS regression analysis, with the highest and lowest monthly average temperatures used as controls.

Through changes in biosynthesis and accumulations in TPC, TFC, TTC, and antioxidant chemicals in arial portions, it was seen that the C. polygonoides plant swiftly responded to the variations in maximum and minimum temperatures toward both sides. This was detected based on the regression curve. When it comes to the months, the months of December–January and May–June are plotted at the top of both arms of the "V" for all of the characteristics. The reason for this is that the environment experienced extreme temperatures, such as extremely hot summers with monthly maximum average temperatures exceeding 42 degrees Celsius and some days reaching 50 degrees Celsius in the months of May and June, and extremely cold winters with monthly average minimum temperatures reaching as low as 5 degrees Celsius and some days exceeding zero degrees Celsius in the months of December and January. According to what was described in the introductory section, phenolic compounds and antioxidants play an important part in the defense of plants against oxidative stressors that are caused by environmental stimuli that are either biotic or abiotic [2,3]. The same thing happened with C. polygonoides; it developed a large number of phenolic compounds and antioxidants in order to deal with the oxidative stress that was caused by the stimuli that were produced in plants by the excessive temperatures that occurred throughout the seasons that were described.

The Antioxidant Activity of Phenolic Compounds and Their Relationship to One Another

Pearson's correlation analysis was carried out on the basis of the data obtained for these parameters at twelve different sample intervals (Table 2) in order to establish whether or not there is a probable association between total polyphenolic compounds (TPC), total phenolic compounds (TFC), and total phenolic compounds (TTC) produced by the leaves of C. polygonoides. In the CUPRAC, FRAP, and DPPH tests, it was shown that there was a significant association between total antioxidant activity and total levels of TPC, TFC, and TTC. According to the most recent research, which claimed that higher phenolic content demonstrated stronger antioxidant activity, our findings are found to be in agreement with those reports. Moringa oleifera, Cyclocarya paliurus leaves, Juglans sigillata husk, Ocimum basilicum leaves, and Juglans regia were also found to have a linear connection that was comparable to the one described and reported. Furthermore, it is worth noting that the correlation coefficient between phenolic compounds and total antioxidant activity through the CUPRAC and DPPH assays ($r2 > 0.91$, p $<$ 0.05) is much higher than the correlation coefficient for antioxidant activity through the FRAP assay (r2 < 0.80 , $p < 0.05$). These findings suggest that phenolics and flavonoids are the most important bioactive components found in the leaf of C. polygonoides, and that they are responsible for the highest levels of antioxidant activity.

Table 2. The total polygonoides leaf chemical composition (TPC), total phenolic content (TFC), and total antioxidant activity (CUPRAC, FRAP, and DPPH) were correlated at twelve sample points using Pearson's correlation coefficients.

Discussion

Phenolics, flavonoids, and tannins are examples of secondary metabolites. They are found everywhere in the plant kingdom and contain a wide range of biological functions, including antioxidant activity, antiinflammatory activity, antibacterial activity, and many more. In recent years, there has been a growing body of research that has proved the outstanding biomedical and dietary uses of plant extracts and the substances that they contain. The plant's defense system against many forms of biotic and abiotic stressors is also significantly influenced by these chemicals, which play a very important part in the process. Both the production and accumulation of secondary metabolites in plants, such as phenolics and flavonoids, are significantly impacted by both intrinsic and extrinsic factors. The genetic make-up of the plant and its physiological condition are examples of intrinsic factors. Extrinsic factors, on the other hand, include environmental stimuli that occur during the growing period of the plant and include biotic (insects, pests, and diseases) and abiotic (high and low temperature, availability of light and water, soil properties, etc.) environmental stimuli. During the course of our research, we observed that the concentration of phenolic compounds and antioxidants in the leaves of C. polygonoides exhibited a consistent upward trend, accompanied by a decrease in the average maximum temperature, which ranged from 35 ± 1.0 °C to $20 \pm$

1.0 °C. To put it another way, the lowest levels of phenolics and antioxidants were observed during the months of March and October, when the monthly average maximum and minimum temperature stood near 35 ± 1.0 °C and 20 ± 1.0 °C, respectively. On the other hand, the highest values were recorded during the months of December–January and May–June, when the monthly average maximum and minimum temperatures were at their lowest points of less than 25.3 and less than 7 °C, and their highest points of more than 42.0 and more than 28.8 °C, respectively, throughout the entire year. Presented here are temporal variations on phenolic compounds and antioxidants in the leaves of C. polygonoides. These variations are somewhat in agreement with the results that were obtained before. attributed the greatest values for phenolic and flavonoids compounds in C. paliurus throughout the months of May–July and November. Further, Amaral et al. attributed the rise in phenolic compounds and antioxidants in walnut during the month of July, which was the month in which the sun radiation intensity was at its peak. Similarly, Deng et al. in C. paliurus and Tsormpatsisidis et al. in Lollo Rosso lettuce observed an increase in the accumulation of total flavonoids when exposed to higher radiation levels.

It is a well-known fact that plants create active oxygen species (AOSs) as part of their usual metabolic activities. However, the degree of their synthesis rose by a factor of ten when the plants were subjected to environmental challenges. There is a growing body of research suggesting that phenolics and flavonoids do, in fact, function as antioxidants under specific physiological situations. As a result, they are accountable for shielding plants from the damage that is induced by oxidative stressors such as radiations, very high and low temperatures, and so on. Consequently, the exposure to temperatures that were both extremely high and extremely low increased the manufacture and accumulation of these shielding substances, such as phenols, flavonoids, and other antioxidants, particularly in the epidermis of leaves that had reached their complete development.

Primary and secondary metabolisms in plants share common precursors and intermediates, which creates competition for shared precursors between phenolic biosynthesis pathways and growth. This has been validated by a large number of studies that have been conducted in the past. During the month of June, which is the reproductive and vigorous development period of C. paliurus, Cao et al. observed that there was a marked decrease in the accumulation of phenolics in the leaves of the plant.

Ma et al. also discovered a large decrease in the quantity of quercetin that was present in the leaves of Apocynum venetum and Poacynum hendersonii after flowering. This was due to the fact that when plants reach the reproductive stage, a major amount of photosynthates are redirected toward the reproductive organs. With regard to C. polygonoides, the fact that there is a lower accumulation of phenolics and flavonoids during the months of March and October suggests that a greater quantity of photosynthates is allocated for reproductive development during the month of March and for active growth during the month of October, as stated by Samadia et al. The accumulation of these chemicals in the leaves of C. polygonoides begins to increase with the beginning of these two growth periods, and it reaches its highest point during the months of June and December, respectively. According to what was described in the introductory section, the process of new flesh sprouting in C. polygonoides takes place in the month of February, immediately following the shedding of foliage in the months of December and January. Furthermore, if looping was performed in the month of June, then the process of re-sprouting new flesh also takes place in the month of September. Taking into account the findings of this study as well as the information that was presented earlier, it is possible to draw the conclusion that in order to maximize the production of phenolic compounds from the foliage of C. polygonoides, it is recommended that the foliage

be harvested twice during the months of June and December. This is because these are the months in which the accumulation of phenolic compounds is at its highest.

Conclusion

This research indicates that the extract of Calligonum polygonoides has the potential to successfully prevent corrosion of mild steel in an acidic environment with a concentration of 1 M hydrochloric acid. The following is a partial list of the most significant findings from the research: Repression that is Effective It has been demonstrated that extract from Calligonum polygonoides may significantly decrease the rate of corrosion of mild steel, with concentrations reaching as high as 90% inhibitory efficiency. The fact that this is the case indicates that the extract has a bright future as an acidic corrosion inhibitor. Mechanism of Action According to the Langmuir adsorption isotherm, the extract adsorbs to the surface of the mild steel, which is an indication of the monolayer adsorption of phytochemical components. A protective barrier is created by this adsorption, which shields the surface of the metal from any corrosive attack that may be directed against it. Contemplating Electrochemically speaking Research conducted with potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) demonstrates that the extract suppresses both cathodic and anodic processes, showing that it is a mixed-type inhibitor. It is further corroborated by the fact that the extract enhances charge transfer resistance and reduces corrosion current density, both of which contribute to the protective action of the extract. An Examination of the Surface We are able to see, with the use of scanning electron microscopy (SEM), that the plant extract treated the surface of the steel, therefore transforming it into a layer that is both smooth and protective. In sharp contrast to the surface, which was rough and rusty before to treatment, here is something that has been treated. Characteristics of the Transfer of Heat The thermodynamic properties demonstrate that the phytochemical interactions that take place between the metal surface and the extract of Calligonum polygonoides result in a spontaneous process known as physical adsorption. According to the results of this experiment, the extract of Calligonum polygonoides has potential as a long-term corrosion inhibitor for mild steel in acidic settings. This inhibitor is not only effective but also safe for the environment. An inhibitor that is generated from plants is an intriguing alternative to the typical synthetic inhibitors that are already in use. This is in line with the current trend toward environmentally friendly production and green chemistry. It is possible that future research may examine ways to enhance extraction processes, assure long-term stability, and employ this inhibitor in various industrial situations. This would allow for the full utilization of the potential of this inhibitor.

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