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α,β-Dehydroamino acids are noncoded amino acids found in natural peptides with interesting biological properties. It is well known that the activity of compounds is determined by their conformational properties. To date, the conformational preferences of dehydroamino acids with nonpolar side chains have been studied and described, in particular dehydrophenylalanine ΔPhe, dehydroalanine ΔAla, dehydrobutyrine ΔAbu, dehydrovaline ΔVal, and dehydroleucine ΔLeu. Dehydroamino acids with polar functional groups also exist in nature, so this Doctoral Thesis examined the conformational properties of β-chlorodehydroalanine ΔAla(β-Cl), O-methyldehydroserine ΔSer(O-Me), and β-aminodehydroalanine ΔAla(β-NH).

A preliminary understanding of conformational preferences was obtained based on the results of computational studies. Potential energy maps E = f(φ,ψ) were constructed for both geometric *Z/E* isomers of each studied dehydroamino acid in three environments (gas phase, chloroform, and water). Possible conformations were then found by identifying local minima. Next, model compounds were synthesized and analyzed by the X-ray structural analysis and spectroscopic studies (NMR, IR), allowing for the assignment of *Z/E* geometric isomerism to the side chain and the identification of the conformations adopted in solution and crystal structure.

It was determined that polar functional groups in the side chain, such as chlorine, methoxy, or amino, influence the conformational properties of dehydroamino acids by forming hydrogen bonds and participating in π-electron coupling, thereby stabilizing certain conformations. β-Chlorodehydroalanine, a structural element of victorins, exhibits different conformational preferences depending on the isomerism of its side chain. The *Z* isomer tends to adopt the α, β, and β2 conformations, while the *E* isomer occurs predominantly in the C5 conformation. In aqueous environments, it can also adopt the β and α conformations. O-Methyldehydroserine, present in cyrmenins, prefers the C5 and β2 conformations in weakly polar environments, while in polar solvents, it readily adopts the α and β conformations. The structural element of callyaerins and callynormine A, β-aminodehydroalanine, tends to adopt the β2 conformation in weakly polar and the α conformation in polar environments.

This paper also proposes new methods for the synthesis of β-chlorodehydroalanine and β-aminodehydroalanine. The first one was obtained by chlorination of dehydroalanine, and the second one by amination of β-bromodehydroalanine. The gained research results allowed for a better understanding of the relationship between the structure of the analyzed dehydroamino acids and their properties. The obtained information can be used in medicine to design new drugs or for agricultural purposes in the development of plant protection products.