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Nature has been recognized as a rich source of useful phytochemicals. Over the years, phytochemical studies have led to the discovery of several natural products, which has a variety of biological activities. As a contribution, this thesis presents the results obtained from the phytochemical study of cyclitols within Polish plants. The study was based on the isolation, purification, identification and quantification of cyclitols using different chromatographic and spectroscopic techniques. The quantities of cyclitol in different morphological parts of *Medicago sativa* L., *Solidago* genus and different morphological parts of *Phacelia tanacetifolia* Benth., which all are species that grow naturally in Poland were determined. In addition, the influence of different factors which can affect the quantities of cyclitols in plant extract were studied, like the part of the plant, the cultivation area, extraction solvent and extraction method. Five different extraction techniques (maceration, Soxhlet, PLE/ASE, UAE, SFE) were applied in this study for isolation of sugars and cyclitols. Among of them, PLE registered the most efficient extraction technique for isolation of sugars and cyclitols from plant materials. On the other hand, the influence of different extraction solvents (96%ethanol, 70%ethanol, water) was also investigated with various extraction techniques. SPE was used as purification and preconcentration method for all obtained extracts. GC-MS and HPLC as sensitive and selective chromatographic techniques were applied for the identification and quantification analysis of cyclitols. In addition, a new fast and sensitive method using MALDI-TOF-MS for identification of cyclitols was developed in this study. Moreover, for selective separation of cyclitols from sugars yeast treatment followed by anion exchange resin and crystallization is an efficient method.