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## Detection of Gamma Irradiated Medicinal Herbs with EPR Method



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### ABSTRACT

In the present work the results of EPR studies on medicinal herbs before and after gamma radiation were investigated. Before irradiation all samples display singlet EPR spectra. After irradiation gamma-induced cellulose free radicals detected in medicinal herbs. Fading study of the radiation-induced cellulose free radicals and heat sensitivity of the central EPR lines in irradiated and non-irradiated samples was detected as a piece of evidence for pre-radiation process. In the light of the findings on all EPR experimental data, we suggest that extending the period for identification of radiation treatment of all medicinal herbs in solid dry state could be technically feasible.



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## INTRODUCTION

For more than 30 years, In 1980, A joint Expert Committee of Food and Agriculture Organization / International Atomic Energy Agency/ World Organization on Food Irradiation (JECFI)[1] concluded "The Irradiation treatment of any food commodity up to dose of 10 kGy exhibit no toxicological hazard; toxicological testing of foods is no longer essential". As a result of these mentioned, ionizing radiation has received acceptance for use in different food types and food industries and has been approved as an effective and promising food safety method. The main concern of irradiation progress made in the commercialization of food irradiation technology is the conformation of irradiation for the applied foods and also loss of detection of irradiation through storage time. At the same time, the regulations of food irradiation treatment differ from country to country, in the future irradiation of foods authorised in some countries and not in other. It is required to designate a analytical method to discriminate between irradiated and nonirradiated food products. When a food is irradiated it is necessary to measure the amount of absorbed dose [2]. There are several physical methods Thermoluminescence (TL), chemiluminescence, Electron Paramagnetic Resonance (EPR), Photo Stimulated Luminescence (PSL) and gas chromatography (GC) have improved for detection of irradiated foods [3-5]. In all of them, EPR is the most leading method and there is continuously interest to investigate radiation-induced signal of various food products [6-16]. This study aims to analyze the behavior of Electron Paramagnetic Resonance (EPR) signals of irradiated some medicinal herbs concerning type, dose-dependency and time stability.

## EXPERIMENTAL

All medicinal herbs (walnut leaves, quince leaves, avocado leaves, hawthorn leaves, chaste leaves, Mate leaves, rowan leaves) were provided from different region of Turkey and stored at room temperature with protected from light and humidity. Samples were dried then powdered for experimental studies. All irradiation were performed at room temperature using  $^{60}\text{Co}$  gamma cell suppling a dose rate of 1.41 kGy/h as an ionizing radiation source at the Sarayköy Establishment of Turkish Atomic Energy in Ankara. The dose rate at the sample sites was measured by a Fricke dosimeter. Determinations were performed on samples irradiated at room temperature with dose range from 0.25 to 1 kGy(0.25,0.5,0.7,1), the permissible dose range for insect disinfestation of food commodities. The EPR spectra of both irradiated and non-irradiated powder samples of medicinal herbs were recorded at room

temperature with a Bruker EMX model spectrometer operating at a microwave power of 0.499 mW, microwave frequency of 9.8 GHz, modulation amplitude of 0.104 mT, and magnetic field modulation frequency of 86 kHz. The g factors were calibrated by comparison with a DPPH sample ( $g=2.0036$ ). Modifications in the resonance line intensities and in the spectrum pattern with microwave power at room temperature were also studied in the range of 0.005-2 mW.

## RESULTS AND DISCUSSION

While non-irradiated all medicinal herbs display very weak EPR singlet characterized with  $g= 2.0048\pm 0.0005$  (Fig 1), gamma-irradiated samples exhibit a strong singlet with the appearance of the triplet structure (Fig 2). This triplet EPR spectrum have pair of satellite lines which settle to the left and right of strong singlet. This appearance can be easily attributed cellulose free radical. The feature of nonirradiated EPR spectra of medicinal herbs probably a superposition of several lines, the occurrence of satellite lines by irradiation is strong proof for the pre-radiation treatment of the samples. It is important to analyse the time stability of radiation-induced cellulose free radicals to discriminate between irradiated and nonirradiated medical products. The samples irradiated at a dose of 10 kGy was studied to determine the effect of storage on the signal intensity of radiation-induced free radicals. Samples were kept in the dark at room temperature over 90 days, the EPR spectra were recorded periodically during this storage time and life time of the radiation-induced free radicals are observed. It was shown that at normal storage conditions the triplet feature of samples disappear within a period of 70-90 days after radiation treatment. Considering that one year is the period in which new fresh medicinal herbs from the next crop are gathered it is needed to extend the application of EPR for distinguishing of irradiated herbs from 90 days to about 180 days. A new approach reported on the potentiality of prolongation of identification period of irradiated cellulose-containing species and herbs by EPR technique[10-13]. Based on this technique non-irradiated and irradiated samples were heated at 60 C for one hour, at four weeks after irradiation. The temperature of 60 C has been selected because at this temperature Maillard reaction may cause paramagnetic species [14-15]. In addition, at this temperature peroxides (ROOR) and hydroperoxides(ROOH) occurred by free radical recombination destruction. The experimental results exhibit that heating gamma irradiated herbs and species to 60 C effected the peak to peak signal intensity of EPR central line. As seen from fig 2 in the non-irradiated samples the decrease of the intensity is

approximately 10%. When the sample was gamma irradiated the decrease was about 50%. This results demonstrated the pre-radiation treatment of studied samples.

### Study of EPR signal decay kinetics

After six months storage period, OSL signal was lost for most of the origin and sample type. At the end of sixth month, an ESR analysis was performed to detect the accuracy of the OSL technique.

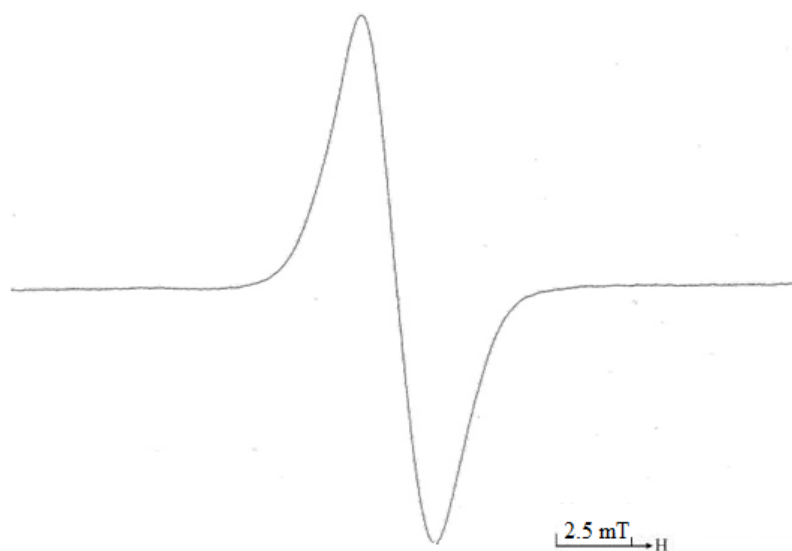
### CONCLUSIONS

EPR spectroscopy is successful method to examine irradiated medicinal plant products even after radiation treatment. With respect to the results of EPR experiments, it was observed that this medicinal herb may be used for prediction of low doses approximately 0.05Gy. Radiation induced EPR signals. The triplet EPR spectrum of cellulose free radical has perfect stability at room temperature approximately 6 months, after that the central singlet EPR signal left, heating the sample to 60 C for one hour caused a considerable decrease in EPR central intensity only in irradiated sample not non-irradiated sample. This procedure unambiguously extended the identification of the interval of previously irradiated studied medicinal herbs. This study perfectly demonstrates that heating pre-irradiated medicinal herbs to 60 C for one hour give rise to substantially.

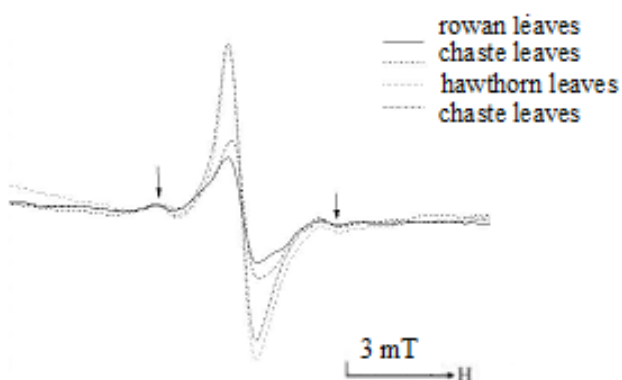
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**Figure 1**



**Figure 2**