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# Gamma Irradiation with 50 kGy Has a Limited Effect on Agronomic Properties of Air-Dry Soil

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**Abstract:** International collaboration on agronomy projects often requires the shipment of soil samples between countries to conduct analyses. However, quarantine regulations in numerous countries restrict the importing of soil samples unless they are sterilized, or analysis is carried out only in quarantine facilities, which greatly increases cost. Yet, sterilization is only an option if it does not change the soil properties. There is conflicting information about the effect of irradiation on soil chemical properties. To assess the effect of gamma irradiation on some soil chemical properties, one hundred randomly selected air-dried (40 °C) soil samples were split into two samples. One sample was left untreated and the other sample was irradiated with 50 kGy as prescribed by Australian biosecurity regulations. Commonly measured agronomic soil chemical properties were then measured and results from the non-irradiated samples were compared to the irradiated samples. The results show no effect of irradiation on soil cation exchange capacity, exchangeable cations, total carbon and nitrogen content, and DTPA-extractable Zn. Small (<5%) but statistically significant effects of irradiation were observed for pH (1:5 water), electric conductivity (EC1:5), DTPA-extractable Cu, Fe and Mn, and Colwell P. The irradiation effects on Fe were greater in the topsoil than subsoil. Considering that irradiation-induced changes to soil chemical properties were below 5%, gamma irradiation can be considered a suitable method to sterilize air-dried soil to meet import requirements, without affecting the interpretation of soil fertility reports.

**Keywords:** quarantine; biosecurity; chemical analysis; soil fertility; sterilization



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## 1. Introduction

International collaboration between agronomists and soil scientists often requires the movement of soil samples across countries or continents to conduct soil analyses. Sometimes, the unavailability of specialized equipment or trained personnel requires shipment of soil to countries where these facilities are available. However, quarantine regulations in countries such as Australia, Canada, New Zealand, Singapore and the USA prescribe that imported soil either needs to be sterilized or used only in quarantine approved facilities.

Quarantine regulations stipulate that soil samples can only be imported by permit holders, staff need training and approval prior to being allowed to work with quarantine samples, and samples must be stored, processed and traced within a designated quarantine approved facility [1,2]. Hence, the administrative costs and management burden associated with the analysis of samples in quarantine-approved laboratory facilities have more than doubled in recent years, compared to non-quarantine samples. Thus, it would be beneficial if samples could be sterilized instead, as these samples are then no longer subject to quarantine and can be analyzed in non-quarantine laboratories. However, a prerequisite is that sterilization of soil does not change the soil properties under investigation.

Approved soil sterilization methods are heat treatment at 160 °C, autoclaving at >120 °C, fumigation, or gamma irradiation with 50 kGy, and a number of reviews have compared the effect of different sterilization methods on soil biological, chemical and physical properties [3–5].

Any heat treatment above 40–60 °C (i.e., equivalent to air drying) changes many soil physico-chemical properties, as interlayer water is driven out of the clay matrix, irreversibly changing soil characteristics such as soil surface area and soil structure [6,7]. Dry heat treatments will also change hydrous metal oxides, and this can affect the availability of elements due to changes in sorption characteristics [8]. Fumigation changes chemical properties of soils, making this method unsuitable for samples destined for chemical analysis [4]. Moist heat treatments are considered to be more detrimental to soil properties than dry heat treatments [3]. Gamma irradiation is considered to be the least disruptive to soil samples intended for physical and chemical analysis [4,9,10].

Treatment with radiation reduces soil biological activity in a dose dependent manner, with 50 kGy considered to kill all (or most) biological activity [3,11]. A review by McNamara et al. [3] on the effect of irradiation reported effective sterilization of soils with >40 kGy, but results for radiation effects on soil chemical properties were contradictory.

For instance, it has been reported that 20–100 kGy gamma irradiation may increase [5,9], decrease [12,13] or not affect [8,10] soil pH. Likewise, micronutrients may increase [5,13,14], decrease [15] or remain unchanged [5,15,16] after gamma irradiation. The effects do not appear to increase with radiation dosage. There is widespread agreement that 30–50 kGy gamma irradiation increases ammonium [9,11,13,17,18] and dissolved organic carbon concentrations [11,13,15,18–20]. This is most likely caused by the lysis of microbial cells as a consequence of gamma irradiation [3,4], Yet, total organic matter [17] and total nitrogen concentrations [21] appear to be unchanged after 30–50 kGy gamma irradiation. Gamma irradiation had no measurable effect on soil surface area and mineralogy [5,12,22] at low doses (20–50 kGy). However, high radiation (>100 kGy) doses may change the oxidation state of iron minerals and surface structure of Si-boride minerals [23,24], and this can affect the adsorption of ions. It should be noted that gamma ray technology for water and bulk density determinations of soils rely on lower energy rays and a lower dosage [25], whereas the energy of gamma rays and dosage for soil sterilization is higher, and is expressed as the absorbed dosage. The absorbed dosage compensates for the attenuation of radiation by the bulk soil, size of container, and water content of the soil.

Some researchers have found that gamma irradiation (30–100 kGy) may affect organic matter rich-soils more than mineral soils [9,17], likely due to degradation of humic organic matter [26]. Thus, some chemical parameters may change depending on the organic matter content of soils, giving rise to contradictory results [3,17].

Overall, the information available on the effects of gamma irradiation on agronomic soil chemical properties is inconsistent. This is possibly due to various irradiation doses, whether wet or dry soil samples are irradiated, and different methods used for chemical analysis. At present, there are no comprehensive and statistically robust studies comparing and evaluating the effect of gamma irradiation on agronomically important soil chemical properties that are routinely measured on air-dried and sieved (<2 mm) soil samples. Past studies only investigated a subset of soil fertility parameters and compared only a few samples. Thus, these studies were not conclusive in determining the effect of gamma irradiation on soil chemical properties.

We have used 50 kGy because this dose is required by regulatory bodies in many countries, for example, Australia, Canada, New Zealand, Singapore and the USA. The aim of this study was to conclusively determine the effect of irradiation at 50 kGy on a large number of soils (100 samples of varying properties), determining an extensive suite of parameters used for soil fertility studies, *viz.* pH in water (1:5 dilution) and electrical conductivity (EC 1:5 dilution), DTPA-extractable plant micronutrients (Cu, Fe, Mn, Zn) and ammonium-acetate exchangeable cations including Ca, Mg, Na, and K, cation exchange capacity (CEC), Colwell extractable P, and total C and N, using a pairwise comparison of irradiated and non-irradiated air-dry soil samples. We did not measure $NO_3$-N and $NH_4$-N pools since these pools change rapidly in moist warm soil and during soil drying. Furthermore, it is only the total N pool that is driving the C:N ratio in soils which determines nitrogen availability to crops. Soil physical or biological properties were also excluded from the comparison since our focus was on soil chemical fertility.

## 2. Materials and Methods

A set of 100 soil samples were collected during three soil surveys conducted in central and northern NSW, Australia (SALIS Survey Numbers 1005215, 1005260, and 1005203) (Supplementary Table S1). Samples comprised the topsoil (A) and subsoil (B, C) horizons, with textures ranging from clay to sandy loam to silty loam. Samples were representative of most soil orders of the USDA Soil Taxonomy [27] (Supplementary Table S1).

The soils were broken into aggregates < 3 cm in diameter during sampling, and transported to the laboratory. Samples were dried at 40 °C for 5–7 days, crushed to pass a 2 mm size mesh, and stored for several weeks at room temperature until analysis [28]; these air-dried samples still contained between <0.5% and 5% water. Drying of soil is required prior to chemical analyses [28,29] and permits long-term storage of soil, e.g., for soil archives [30].

Each sample was then split into two sub-samples of approximately equal mass (ca 100 g) and stored in polyethene bags. One sub-sample was left as-is, whilst the other sub-sample was irradiated by Steritech Pty Ltd. (Narangba, Queensland, Australia). Samples were packed in a cardboard box (approx. 40 × 30 × 15 cm) and placed on a pallet in the irradiation chamber. The irradiation procedure uses a $^{60}$Co gamma radiation source (1.17 MeV and 1.33 MeV lines) and an absorbed dose of 50 kGy was administered based on Harwell Red 4034 Dosimeters (poly-methylmethacrylate (PMMA) sachets) placed inside the pallet; the temperature during irradiation did not exceed 40 °C. The absorbed radiation dose takes into account attenuation due to the bulk of the soil and differences in water content. This gamma irradiation protocol is audited and approved by the Australian Government Department of Agriculture for treatment of quarantine material [31,32].

### 2.1. Soil Chemical Analysis

Soil chemical analyses were carried out on the 100 pairs of soil samples using the same equipment and reference samples for quality control, and using the following standard or ISO methods:

pH: Soil:water ratio of 1:5, shaken for 1 h, allowed to settle for 1 h and pH measured in the stirred sample [33]

EC: Soil:water ratio 1:5, shaken for 1 h, allowed to settle for 1 h, and EC measured on the un-stirred supernatant [34].

Phosphorus (modified Colwell method) 1:100 suspension of soil in 0.5 M Na-bicarbonate (pH 8.5), shaken for 16 h, centrifuged and supernatant analyzed by ICP [35].

Exchangeable Ca, Mg, K, and Na and CEC: Soil suspension (1:20) in 1 M $NH_4Cl$ (pH 7) shaken for 1 h, centrifuged and supernatant analyzed by ICP (Method 15A1 in Rayment and Lyons [36]). The CEC was calculated from the sum of exchangeable cations (including 1 M KCl extractable Al and protons in acid soils).

Total C and N: Samples (~1 g) were placed in porcelain crucibles into a LECO CN combustion analyzer [37,38].

Plant micronutrients Cu, Fe, Mn, Zn: Soil was suspended in 0.005 M DTPA + 0.01 M $CaCl_2$ + 0.1 M TEA solution pH 7.2 (1:2 ratio), shaken 2 h, centrifuged and supernatant analyzed by ICP, according to ISO 14870 method [39].

### 2.2. Statistical Analysis

Exploratory data analysis of the soil parameters with Quantile-Quantile Plots (Proc Univariate in SAS) indicated that the data were not always normally distributed. Therefore, we converted all parameters with the Box-Cox transformation (Proc Transreg in SAS), after which the parameters met the requirements of normality as determined by the Shapiro–Wilk test. Since our interest was to compare irradiated with non-irradiated samples, we used the Bland–Altman approach [40–43] using SigmaPlot v13. For each soil, we determined the difference between the transformed values of the irradiated and non-irradiated sample for each soil property and plotted these against the transformed non-irradiated values. If the 95% confidence interval of the difference did not include zero, irradiation was considered to have a significant effect on the soil property and the probability level was calculated by the paired t-test using Proc Ttest in SAS. Soil parameters which were significantly affected by irradiation were further analyzed using Proc Glm in SAS to determine whether the effect of irradiation could be attributed to either soil horizons, soil textures or soil type.

## 3. Results and Discussion

There were no significant differences between irradiated and non-irradiated air-dry soil samples with respect to the total soil carbon, total soil nitrogen, exchangeable Ca, K, Mg, Na, the CEC, and DTPA-extractable Zn (Table 1). Gamma irradiation with 50 kGy had a slight but statistically significant effect on EC, pH, P, Cu, Fe and Mn (Table 1). For Fe, the irradiation effect differed between topsoil and subsoil samples ($p$ = 0.0174), but irradiation-induced changes in the other parameters were not significantly affected by soil type, horizons or textures (data not shown).

**Table 1.** Effect of 50 kGy gamma irradiation on agronomically relevant soil chemical properties. Shown are the mean, minimum and maximum values of 100 samples before and after irradiation, the percentage difference (non-irradiated = 100%), and the probability that the mean difference is significantly different from zero (paired $t$-test). Properties shown in bold were significantly ($p < 0.05$) affected by irradiation.

| Soil Property | Gamma-Irradiated Samples | | | Non Irradiated Samples | | | Mean % Change | $p$ |
|---|---|---|---|---|---|---|---|---|
| | Mean | Min | Max | Mean | Min | Max | (Min–Max Range) | |
| **pH (1:5, $H_2O$)** | 6.90 | 4.80 | 9.31 | 6.93 | 4.88 | 9.31 | −0.5 (−3.7–+1.8) | **<0.001** |
| **EC (1:5), dS/m** | 0.15 | 0.04 | 1.26 | 0.15 | 0.03 | 1.29 | 3.9 (−17.4–+33.7) | **<0.001** |
| Total C, % | 1.97 | 0.13 | 9.33 | 1.96 | 0.10 | 9.45 | 0.2 (−8.1–+33.4) | 0.346 |
| Total N, % | 0.18 | 0.05 | 0.60 | 0.18 | 0.05 | 0.61 | −0.4 (−16.0–+13.4) | 0.428 |
| **Colwell P, ppm** | 46.96 | 0.63 | 191.72 | 45.06 | 0.74 | 191.1 | 4.2 (−48.0–+43.4) | **<0.001** |
| Exchangeable Ca, $cmol_c$/kg | 13.68 | 0.78 | 34.12 | 13.67 | 0.81 | 34.21 | 0.1 (−9.2–+6.6) | 0.683 |
| Exchangeable K, $cmol_c$/kg | 0.60 | 0.14 | 3.03 | 0.59 | 0.16 | 2.98 | 1.3 (−13.4–+27.7) | 0.255 |
| Exchangeable Mg, $cmol_c$/kg | 6.47 | 0.84 | 27.06 | 6.45 | 0.84 | 27.05 | 0.2 (−9.2–+7.8) | 0.332 |
| Exchangeable Na, $cmol_c$/kg | 0.92 | 0.01 | 11.06 | 0.91 | 0.01 | 10.73 | 1.4 (−85.8–+349.4) | 0.422 |
| CEC, $cmol_c$/kg | 21.67 | 2.24 | 62.59 | 21.63 | 2.24 | 62.38 | 0.2 (−6.9–+5.1) | 0.463 |
| **DTPA Cu, mg/kg** | 1.35 | 0.13 | 3.80 | 1.33 | 0.13 | 3.89 | 1.3 (−12.1–+17.9) | **<0.001** |
| **DTPA Fe, mg/kg** | 30.62 | 1.26 | 228.47 | 31.10 | 1.23 | 232.28 | −1.5 (−15.4–+15.3) | **<0.001** |
| DTPA Zn, mg/kg | 1.34 | 0.09 | 17.91 | 1.33 | 0.10 | 17.70 | 0.1 (−21.5–+28.6) | 0.985 |
| **DTPA Mn, mg/kg** | 69.28 | 1.33 | 223.92 | 68.14 | 1.01 | 216.66 | 1.6 (−25.4–+31.2) | **0.002** |

The changes in EC (+3.9%) and pH (−0.5%) were small, and would be difficult to detect in field trials where spatial and temporal variability in pH and EC is much greater [44,45]. The increase in EC is likely due to lysis of microbial cells after irradiation, releasing the chemicals within the cells into the soil. The change in EC due to irradiation is much smaller than EC fluctuations naturally occurring in soil (e.g., after rainfall or fertilizer application) [46], and we consider the magnitude of change to be of no practical relevance. The effects of radiation on pH in published research are inconsistent, with Bank et al. [12] and Menzies et al. [13] describing decreases in pH by 1.3–18%, while Alphei and Scheu [9] and Wolf et al. [5] found that pH increases by 1.4–9%. The changes in pH observed in this study were low (−0.5%) and would not affect soil and fertility management (e.g., liming recommendations).

After gamma irradiation, Colwell P concentration increased by 4.2%. Alphei and Scheu [9] reported a several-fold increase in Olsen P, whereas Eno and Popenoe [17] found no effect on P, and McNamara et al. [3] cited results showing either a decrease or increase in P. The increase in Colwell P may be due to release from lysed microbes or from organic matter in the soil [47], and the small increase in P after irradiation would not warrant adjusting fertilizer recommendations for P when based on irradiated soil samples.

Irradiation increased the DTPA-extractable plant micronutrients Cu (+1.3%) and Mn (+1.6%), whereas Fe decreased (−1.5%). The observed effects of irradiation were much smaller than effects described by other authors: Staunton et al. [15] and Menzies et al. [13] reported a 2-fold increase in Mn, and Salonius et al. [14] recorded a 60–80% increase in Mn and Cu. On the other hand, Gore and Snape [16] found only very small changes in acetate-leachable macro-and micronutrients after 50 kGy gamma irradiation of dry soil, in agreement with our results. The slight changes in DTPA-Cu, Fe, and Mn with irradiation may be due to effects on some soil mineral constituents. For instance, Bank et al. [12] found that 20 kGy gamma radiation increased sorption of $U^{(VI)}$ ions by increasing divalent iron in soil and Krausse et al. [8] suggested that irradiation with 55 kGy changed hydrous metal oxides, thereby affecting adsorption processes in soil. It has been proposed that gamma irradiation forms OH• and H• radicals which impart oxidizing or reducing properties [4] and reducing conditions generally increase availability of Mn and Fe [48]. The irradiation effect on extractable Fe was greater on the topsoil horizon (−2.20%) than the subsoil horizon (−0.14%) and this may be attributed to the greater presence of Fe-organic matter complexes in topsoil. This aspect would require further investigation as it implies that soils rich in Fe-complexes (e.g., Spodosols) may be affected by gamma irradiation. Our soil dataset did not contain Spodosols (Supplemental Table S1).

Despite the lack of irradiation effect on total C and N in the soils examined, it is important to note that irradiation may affect C and N pools [3] due to lysis of microbial cells which leads to an increase in soluble N and C [11,13,17,18,20]. We quantified only total C and N, rather than identifying individual pools, since the total C:N ratio is the important indicator of N availability in soils. As soil C and N pools change rapidly in moist soil, we chose air dried soils. Otherwise, soils need to be frozen immediately after sample collection [6,49], which is difficult in developing countries, or when sampling in remote locations. Shipping frozen and field-moist soil incurs greater shipping costs and poses logistical challenges. In addition, all routine agronomically relevant soil properties are determined and expressed on air-dry soil basis. In addition, soil archives store dry soil, and NIST reference soil samples are supplied dry [30,36]. Air-dry soil is less affected by gamma irradiation than moist soil [3]. However, storage of dry soil at room temperature may still cause changes over time in soil chemical properties such as pH and extractable Ca and Mg [49].

Our results validate those reported by Gore and Snape [16] for trace elements, showing little to no effect of irradiation, but other researchers report large changes. The inconsistency may be due to several factors. Firstly, the residual moisture content of the soils, with moist soils more susceptible to irradiation-induced changes than dry soil [3,18]. Secondly, drying of soil also leads to changes in soluble C and N pools [18] and extractable macro- and micronutrient [6], confounding the effect of irradiation. Thirdly, organic matter rich soil was claimed to show a greater change to irradiation than soil with low organic matter [17]. However, we found no strong correlation between total organic carbon content (ranging from 0.1 to 9.4%) and irradiation-induced changes in soil properties, with correlation coefficients ($r$) ranging from 0.28 to −0.30. Finally, further research would be required to determine if freshly dried soil shows a different response to gamma irradiation than dry soil stored for longer periods.

Many papers reported effects of irradiation on only 2–3 soil samples and this may give misleading results; in our study, we investigated 100 soil samples and found only minor effects. Indeed, a Power Analysis with a 10% margin of error suggested that minimum sample sizes of 62–104 samples are required to draw statistically robust conclusions on the effect of irradiation. The ranges of concentrations of nutrients in our samples were not dissimilar to the ranges reported in literature dealing with irradiation effects (Supplemental Figure S1). Thus, we consider it unlikely that disagreement on the effect of irradiation is due to different concentration ranges between samples and between studies. Rather, we speculate that the storage of soil prior to irradiation may have an effect, as Rechcigl et al. [6] found that all methods of sample storage and processing resulted in changes. It is possible that dry soil stored for longer periods (e.g., few weeks, as in our case) is less affected by irradiation than freshly collected and dried soil. Collecting soil at the beginning and end of field trials, drying the soil and then shipping by surface freight mail means that the soil samples will have "aged" for some time. Likewise, soil libraries and reference soils are stored for extended periods. These soils are probably less likely to undergo changes during irradiation. By comparison, freshly collected soil samples may undergo greater changes during drying and these changes can be compounded by irradiation

## 4. Conclusions

We selected 100 soils that spanned a wide range of chemical and textural properties, ranging from sandy to clay soils, from low total C (0.1%) to high total C (9.45%), from acid (pH 4.9) to alkaline soils (pH 9.3), and from poorly fertile to highly fertile soils. Nevertheless, sterilization of air-dry soil using 50 kGy gamma irradiation had no effect on total C, total N, exchangeable Ca, Mg, K, Na, CEC, and Zn, and small effects on EC (+3.9%), pH (−0.5%), Colwell P (+4.2%), Cu (1.3%), Fe (−1.5%) and Mn (1.6%). Considering the inherently high spatial and temporal variability in soil properties in the field, the effect of gamma irradiation on soil properties is small. Therefore, gamma irradiation can be used to treat imported soil samples to avoid quarantine restrictions, without affecting the interpretation of soil analytical reports.

**Supplementary Materials:** The following are available online at https://www.mdpi.com/article/10.3390/soilsystems5020028/s1, Figure S1: Ranges (minimum to maximum) of values for various soil chemical parameters in studies reporting irradiation effects on soil (shown with red lines) compared to the range in values for the soils used in this study (shown with black lines), Table S1: Complete data set.

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