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Article

Effect of waste mica on transfer factors of ^{134}Cs to spinach and lettuce

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ABSTRACT **A greenhouse pot culture experiment was conducted to study the effect of graded levels of waste mica (0, 10, 20 and 40 g kg^{-1}) on reducing the radiocesium uptake by spinach (*Spinacia olerecea L*) and lettuce (*Lactuca sativa L.*) grown in ^{134}Cs-contaminated (at 37 k Bq kg^{-1} soil) Inceptisols, Vertisols and Ultisols. The biomass yield, and potassium content and its uptake by crops have been significantly improved by waste mica application. The crops grown in Vertisols recorded higher biomass yield, and K content and its uptake as compared with Inceptisols and Ultisols. The average ^{134}Cs transfer factor values recorded were : 0.21, 0.17 and 0.26 at the first cutting, 0.15, 0.12 and 0.28 at the second cutting and 0.07, 0.05 and 0.23 at the third cutting from Inceptisols, Vertisols and Ultisols, respectively. Waste mica significantly suppressed radiocesium uptake, the effect being more pronounced at 40 g mica kg^{-1}soil. There exists an inverse relationship between the ^{134}Cs transfer factors with plant potassium content and also the K uptake by the crops.**

Keywords: ^{134}Cs / potassium / radiocesium / transfer factor / waste mica / spinach / lettuce

RÉSUMÉ **Effet de déchets de mica sur les facteurs de transfert du ^{134}Cs à l'épinard et la laitue.**

Des expériences de culture en pot sous serre ont été conduites afin d'étudier l'effet de concentrations croissantes de déchets de mica (0, 10, 20, 40 g kg^{-1}) sur la diminution de l'adsorption de césium par l'épinard (*Spinacia olerecea L.*) et la laitue (*Lactuca sativa L.*) poussant sur des Inceptisols, Vertisols et Ultisols contaminés par du ^{134}Cs (37 k Bq kg^{-1}). Le taux de croissance de la biomasse, le contenu en potassium et son adsorption par les plantes récoltées ont été significativement améliorés par l'ajout de waste mica. Les plantes ayant poussé sur les Vertisols ont eu les taux de croissance de la biomasse, le contenu en potassium et son adsorption supérieurs à ceux des plantes ayant poussé sur Inceptisols et Ultisols. Les valeurs moyennes des facteurs de transfert du ^{134}Cs ont été respectivement : 0,21, 0,17, 0,26 à la première récolte, 0,15, 0,12, 0,28 à la seconde récolte et 0,07, 0,05, 0,23 à la troisième récolte sur les Inceptisols, Vertisols et Ultisols. Le waste mica a réprimé de façon significative l'adsorption de radio césium, cet effet étant le plus prononcé pour une concentration de 40 g de mica kg^{-1} de sol. Il a été montré que les facteurs de transfert en ^{134}Cs sont inversement proportionnels au contenu en potassium de la plante et à son adsorption par la plante.

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1. Introduction

Entry of radionuclides into the soil environment assumes special significance from the human health point of view, since these can enter the human body via the food chain and increase the radiation risks over a long period. Radionuclides have been introduced into the terrestrial environment by nuclear weapon testing, accidental release from nuclear facilities (*viz*. Chernobyl accident, and Fukushima, Japan), nuclear wastes, etc. Several factors appear to be of paramount importance in the eventual transfer of radiocesium from soil to plants. Such factors include soil type and properties (CEC, specific sorption properties, and the nature and amount of organic and mineral matter) (Manjaiah *et al.,* 2003; Lembrechts, 1993; Monira *et al.,* 2005), radionuclide interaction with the soil, level of potential sorption-competitive species in the soil solution, and type of crop. In addition to the radionuclide fraction available in the soil, the CEC of the soil, the solid-liquid distribution coefficient (K_d) and levels of competitive species (K^+ and NH_4^+) for radiocesium can be used for prediction purposes (Absalom *et al.,* 2001; Fuhrmann *et al*., 2003).

Radiocesium (^{137}Cs) is one of the most important radionuclides, with a long half-life (30 years); its bioavailability has been studied with respect to potassium competition, as potassium is generally considered as an effective inhibitor of radiocesium uptake. Two hypotheses have been proposed to explain the genotypic variation in radiocesium uptake by plants (Keum *et al.,* 2007). They are: (i) the Cs/K discrimination factor is equal for different plants grown in identical conditions, and (ii) the genotypic variation in growth rate and in root surface area results in different radiocesium levels in soil-grown plants. According to the first hypothesis, genotypic differences in potassium levels will correlate with differences in radiocesium levels. The second hypothesis is based on the fact that the Cs mobility in the soil is low; hence the Cs flux to the roots can be below their cesium interception potential. In this scenario, radiocesium levels in the plant are strongly influenced by the growth rate of the plant (through the growth dilution effect) and the root surface area (Keum *et al.,* 2007).

Agricultural countermeasures such as use of counter-ions of the same homologous series, *viz.,* stable cesium, potassium or ammonium, were considered to be more useful in checking the radiocesium transfer to crops (Lembrechts, 1993; Nisbet *et al*., 1993; Zhu *et al.,* 2002; Sandeep and Manjaiah, 2008; Sandeep *et al*., 2009). Clay minerals, micas and zeolites have been observed to reduce the phytoavailability of radiocesium to crops (Paasikallio, 1999) and the effectiveness of such amendments varies significantly depending on plant species and soil parameters (Frissel *et al*., 2002; Sachdev *et al.,* 2006; Willey *et al*., 2005; Broadley and Willey, 1997).

India has large deposits of mica. A huge quantity of waste mica, a by-product of mica industries, with variable K content (8–12%) and crystallinity, is available for use for different purposes. It can be used as an alternate source of potassium if modified or altered by some suitable chemical or biological means (Basak and Biswas, 2009). It can also be used as an amendment to check the cesium transfer from soil to crops. Thus, waste mica can play a duel function, as a sorbent of radiocesium and can also regulate the cesium bioavailability in soils through the Cs-K counter-ion effect. Potassium release from mica is a slow process and greatly controlled by soil and plant parameters. More than 90% of the added radiocesium can be retained by waste mica, and its sorption-desorption was greatly controlled by the particle size of mica (Sreenivasa Chari, 2010).

The present study evaluates the effectiveness of waste mica in minimizing the ^{134}Cs transfer from artificially contaminated soils to spinach and lettuce. ^{134}Cs was used in the present study in place of ^{137}Cs, because of its short half-life (2 years). Spinach and lettuce were used as test crops. Being leafy vegetables, the whole aboveground plant part will be consumable by human beings. In the eventuality of radiological emergency, the total radiocesium load on such crops and its entire transport to human beings can be simulated.

2. Materials and methods

2.1. Description of soils

Three soils (representing Inceptisols, Vertisols and Ultisols, USDA system of soil classification) differing in texture, soil organic carbon, potassium content and clay mineralogical composition (Tab. I) were used for the greenhouse pot culture experiment. The soils were analyzed following standard methodologies. pH in soil water suspension (1:2.5) was measured using a combined electrode (glass and calomel) in a digital pH meter. The electrical conductivity was measured in the supernatant liquid of the soil water suspension (1:2.5) with the help of a conductivity bridge, expressed in dS m^{-1} at 25 °C. Normal sodium acetate (pH 8.2) was used to determine the cation exchange capacity of soils. Organic carbon was determined by the wet digestion method of Walkley and Black (1934) as described by Jackson (1973). Total carbon content in soils was determined by the wet oxidation method (Snyder and Trotymov, 1984). Exchangeable potassium was estimated using a flame photometer after extracting the soils with neutral normal ammonium acetate (Jackson, 1973). Non-exchangeable K was extracted by boiling with 1 N HNO_3 and potassium was estimated by flame photometer (Wood and De Turk, 1941). The clays (<2 µm) were isolated by removing organic matter, sesquioxides and allophones. Basally oriented clay samples (Mg-air, Mg-glycerol,

TABLE I
Salient properties of the soils used in the greenhouse pot culture experiment.
Propriétés caractéristiques des sols utilisés dans les expériences de culture en pot sous serre.

Soil property	Inceptisols	Vertisols	Ultisols
pH (1:2.5) (soil: water)	8.40	8.20	5.50
EC (dS m^{-1})	0.24	0.22	0.05
Organic carbon (%)	0.56	0.64	0.40
Available K (g kg^{-1})	107.1	183.0	56.3
NH_4-N (g kg^{-1})	12.0	15.5	9.3
CEC (C mol (p^+) kg^{-1})	14.95	47.56	6.78
Total organic carbon (%)	1.24	1.42	0.93
Mechanical composition			
Sand (%)	42.00	27.60	45.60
Silt (%)	42.50	17.28	37.28
Clay (%)	15.50	55.12	17.12
Soil texture	Silty loam	clay	Silty loam
Clay mineralogical composition	Mica (51%)	Smectite (88%)	Kaolinites (89%)
	Kaolinite (34%)	Kaolinite (7%)	Mica (7%)
	Vermiculite (15%)	Mica (5%)	Chlorite (4%)

K-air-dried, K-300 °C and 550 °C heated) were X-rayed. The X-ray diffractograms were recorded in a Philips diffractometer (Model 1140) using Ni-filtered Cu-Kα radiation at a scanning speed of 2° 2θ per minute. Identification and semi-quantitative estimation of clay minerals was also carried out (Jackson, 1976; Manoj Kumar *et al.*, 2002).

2.2. Waste mica

Waste mica, a potassium-bearing mineral, was collected from the surroundings of mica mines located in the Koderma district of Jharkhand, India. The waste mica (dominantly muscovite) is generated during the dressing of raw mica blocks, which are generally used as electrical insulators. The waste mica was ground and passed through a sieve to obtain uniform particle sizes of 2 mm. The ground waste mica contained 10.0% total K and traces of water-soluble K. The exchangeable and non-exchangeable K in the waste mica were estimated as 157.5 mg kg^{-1} and 260.0 mg kg^{-1}, respectively. In each experimental pot waste mica was uniformly mixed with the soils, pre-contaminated with ^{134}Cs at 37 Bq kg^{-1} soil. Recommended levels of nutrients were added to pots at the time of sowing/transplanting of crops.

2.3. Growing of the crops

The soils in each glazed pot (30 cm height and 20 cm diameter), holding eight kg of soil, were contaminated with ^{134}Cs radionuclide at 37 kBq kg^{-1} soil. ^{134}Cs in the form of CsCl was obtained from the board of radiation and isotope technology (BRIT), Mumbai, India. The experiment was conducted during the winter season of 2008. In each of the pots, the waste mica as per the levels was uniformly mixed with the soils pre-contaminated with ^{134}Cs. Nutrients as per the recommended schedule of N:P:K (54:27:27 mg kg^{-1} soil) were added through urea, diamonium phosphate and muriate of potash (KCl), respectively. The seeds of spinach (Pusa Bharti) and seedlings of lettuce (Chinese yellow) were sown / transplanted. Ten days after germination, seedlings were thinned out to maintain four plants per pot. The K input through the treatments is shown in the following table. The experiment consisted of 72 pots with the following treatment combinations: (i) two crops, (ii) three soils, (iii) four mica treatment levels: 0, 10, 20 and 40 g mica kg^{-1} soil, and (iv) three replications (2 × 3 × 4 × 3 = 72). The experimental layout followed was a factorial completely randomized design (CRD).

Treatment	**K added through fertilizer (mg kg^{-1} soil)**	**K added through waste mica (mg kg^{-1} soil)**	**Total K input (mg kg^{-1} soil)**
Control	27	0	27.0
Mica (10 g kg^{-1})	27	157.5	184.5
Mica (20 g kg^{-1})	27	315.0	342.0
Mica (40 g kg^{-1})	27	630.0	657.0

Crops were harvested by cutting samples at 5 cm above the soil surface at each cutting so as to regenerate the biomass. The first cutting of crops was carried out after 45 days of sowing. The plants were allowed to grow for another 30 days, then the aboveground plant parts were cut again. After the second cutting, plants were further allowed 30 more days and then the third cutting of samples was collected (105 days after sowing).

2.4. Analysis methods

After harvesting the aboveground plant samples, they were dried initially in the shade for 2–3 days followed by oven drying at 70 °C, and the dry matter yield was recorded separately. The oven-dried plant samples were then ground in a Willey mill using a 20-mesh sieve and stored for further analysis. ^{134}Cs activity and potassium content were measured in these ground and homogenized plant samples. Potassium in plant tissues was analyzed in a triacid digest

(H_2SO_4:HNO_3:$HClO_4$: 9:4:1) by flame photometer. After plant sampling at each stage, about 5–10 g soil was scooped out from each pot, air-dried, ground in a pestle and mortar (100-mesh size), and used for measurement of ^{134}Cs activity and potassium (Jackson, 1973).

The dried and ground plant samples were used for measurement of ^{134}Cs using a 2.5" × 2.5" NaI (TI) well-type detector installed in a 15-cm-thick lead shield and a single-channel gamma analyzer. Similarly, the ^{134}Cs activity in soil samples (100-mesh size) was also counted directly. The ^{134}Cs activity standard prepared on the day of its use for contaminating the soils was continuously maintained and counted at every time of the counting of the activity in soil/plant samples. The activity data in soils and plant samples was used to calculate the soil-to-spinach and lettuce crop transfer factor (TF) as: (Bq kg^{-1} dry plant tissue) / (Bq kg^{-1} dry soil).

The experimental data was statistically analyzed following standard statistical methods (Gomez and Gomez, 1984). For the analysis of the data, a factorial completely randomized design (CRD) was used to evaluate the effects of three factors, *i.e.*, crops (two), soils (three) and treatment levels (four). The F-test was carried out to test the significance of the treatment differences and the least significant difference (LSD) was computed to test the significance of different treatments at 5% of the probability by MSTATC (version 7.0).

3. Results and discussion

3.1. Biomass yield

As compared with the control treatment receiving the recommended level of fertilizers, waste mica application significantly ($p < 0.05$) increased biomass yield of crops. Spinach grown in Vertisols showed consistently and significantly higher biomass yield at all three cuttings (Fig. 1). Crops grown in Vertisols recorded about 1.2 times higher biomass yield as compared with the rest of the soils. Mean values of biomass yield were significantly improved (an increase of 1.5 times) with the highest mica treatment (40 g mica kg^{-1} soil) as compared with control. The interaction effect of crops and soils was significant at the 2nd and 3rd cuttings, whereas the interactions between crops and amendment levels, and soils and treatment levels were found to be significant at all three cuttings. In general, the biomass yield was much higher at the final harvesting stage as compared with the first two samplings.

Both the crops responded positively to the fertilizers; however, the effect was slightly higher in Vertisols, owing to their inherently higher fertility status. Slow and continuous supply of K through the reactivity of the added mica with the

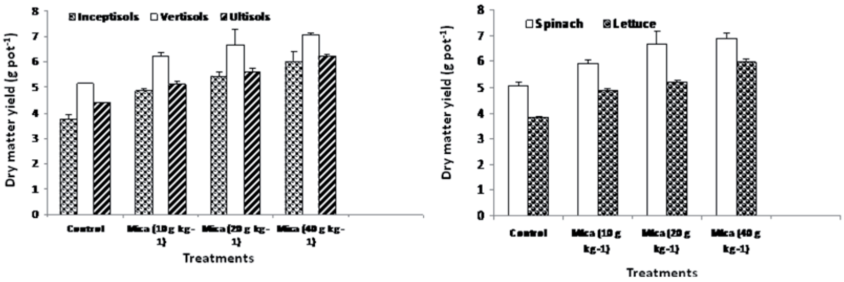

Figure 1 – ***Dry matter yield (g pot^{-1}) in spinach and lettuce as influenced by waste mica.***
Effet de déchets de mica sur le taux de croissance en matière sèche (g pot^{-1}) chez l'épinard et la laitue .

rhizosphere processes further improved the biomass yield of crops. A variety of organic weathering agents arising from decomposition of plant and animal residues, root exudates, organic acids, and organic metabolites from the rhizosphere have been reported to be responsible for mineral weathering/alterations (Boyle and Voight, 1973; Barker *et al*., 1997; Song and Huang, 1988; Basak and Biswas, 2009).

3.2. Potassium content and its uptake

In general, addition of waste mica significantly improved the plant potassium content (Fig. 2). Irrespective of the sampling stages, K content in lettuce was observed to be slightly higher (by 1.2 times). Amongst the soils, crops grown in Vertisols and Inceptisols showed higher K content as compared with Ultisols.

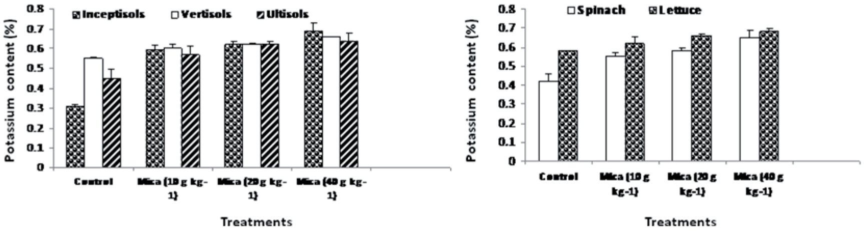

Figure 2 – ***Potassium content (%) in spinach and lettuce as influenced by waste mica.***
Effet de déchets de mica sur le contenu en potassium (%) chez l'épinard et la laitue.

Potassium content was about 1.5 to 1.9 times higher in plants receiving the graded levels of waste mica. The interaction effects between soil and treatments, and crop and treatments were found to be statistically significant at all three stages of sampling.

There exists an inverse and positive relationship between the ^{134}Cs transfer factors with plant %K concentration and also the K uptake by the crops (Fig. 3). Potassium concentration in the growth media is an important factor influencing ^{134}Cs absorption and movement within plants because of its similar chemistry (Lasat *et al.,* 1997; Buysse *et al.,* 1996). Plants grown in K-treated soils showed decreasing uptake of radiocesium (Rosen 1991; Strandberg and Johansson, 1998). A similar relationship was also observed between ^{134}Cs activity concentration in plant tissues and %K content.

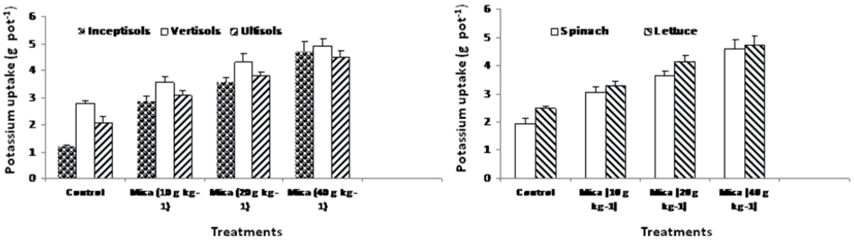

Figure 3 – ***Potassium uptake (×10^{-2} g pot^{-1}) by spinach and lettuce as influenced by waste mica.***
Effet de déchets de mica sur l'adsoption du potassium (×10^{-2} g pot^{-1}) par l'épinard et la laitue.

In general, graded levels of waste mica application significantly improved the potassium uptake by crops as compared with control. Potassium uptake by crops varied from 1.17 × 10^{-2}–4.74 × 10^{-2} g pot^{-1} (at the 1st cut); 1.08 × 10^{-2}–3.09 × 10^{-2} g pot^{-1} (at the 2nd cut) and 1.30 × 10^{-2}–4.60 × 10^{-2} g pot^{-1} (at the 3rd cut) (Fig. 4). Irrespective of the treatments, potassium uptake by lettuce was highest at the first sampling; however, at the 2nd and 3rd cuttings, spinach showed higher values. The general trend of K uptake by crops was observed in the following sequence: Vertisols > Inceptisols > Ultisols. The interaction effect between crops, soils and amendment levels was observed at all the sampling stages. Nearly 1.8 to 2.2 times higher K uptake was observed in treatments receiving the highest waste mica level (40 g kg^{-1}).

Spinach and lettuce, both being exhaustive leafy vegetable crops, responded well to the fertilizer and waste mica application. Graded doses of mica application in general increased the dry matter yield, %K and also the K uptake by crops at all

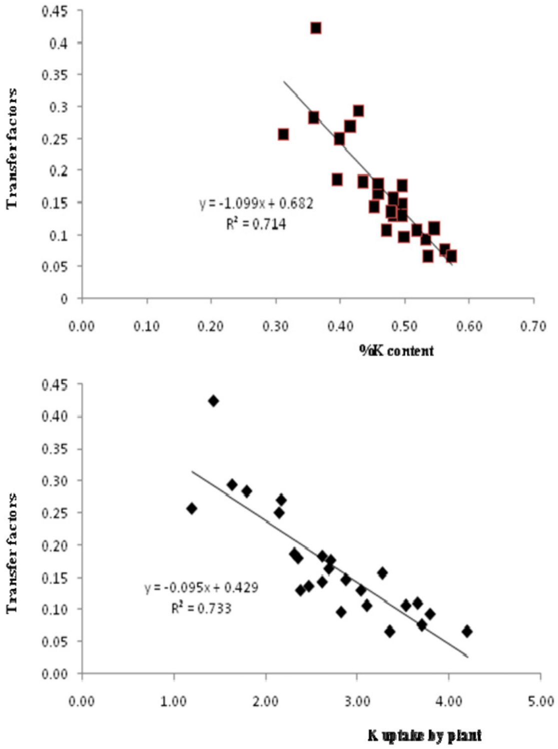

Figure 4 – ***Relationship between ^{134}Cs transfer factors with potassium content and Kuptake by spinach and lettuce.***

Relation chez l'épinard et la laitue entre le facteur de transfert du ^{134}Cs et la concentration en potassium et l'adsoption du potassium.

three sampling stages. Continued release of K^+ ions from waste mica through root activity and the soil K data supports this observation. Vertisols and Inceptisols, owing to their better fertility status, responded well to mica application and enhanced these parameters. Potassium-solubilizing microorganisms are capable of solubilizing the unavailable forms of potassium in K-bearing minerals such as mica, illite and orthoclase through production and excretion of organic acids such as citric, oxalic and tartaric acids. Organic acids can facilitate the weathering of minerals by releasing K from rocks or through the formation of metal-organic complexes by forming chelate with silicon ions to bring the K into solution (Song and Huang, 1988; Calvaruso *et al.,* 2006; Bakker *et al.*, 2004; van Scholl *et al.*, 2008). Other possible hypotheses/mechanisms to mobilize the soil K reserve are due to biofilm formation on the rhizospheric mineral surfaces by certain bacterial strains (Balogh-Brunstad *et al.*, 2008).

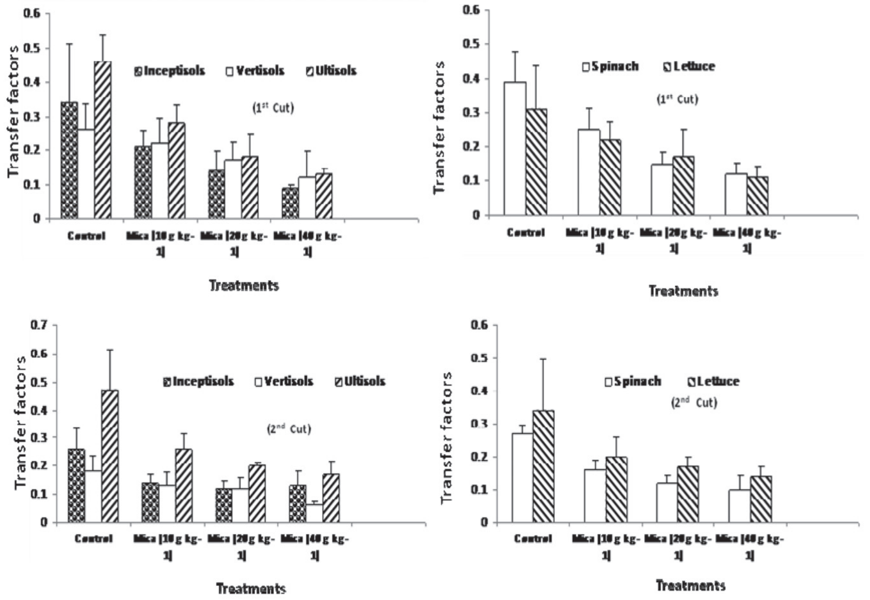

Figure 5 – ***Radiocesium transfer factors in spinach and lettuce as influenced by waste mica.***
Effet de déchets de mica sur le facteur de transfert du radio césium chez l'épinard et la laitue.

3.3. ^{134}Cs Transfer factor

On average, ^{134}Cs transfer factors were 0.21, 0.17 and 0.26 at the first cut; 0.15, 0.12 and 0.28 at the second cut, and 0.07, 0.05 and 0.23 at the third cut for Inceptisols, Vertisols and Ultisols, respectively (Fig. 5). Crops grown in Ultisols showed higher TF, while clayey soil (Vertisols) had the lowest transfer factor values. These are comparable with the reported mean TF values from sub-tropical environments ($6 \times 10^{-2} \pm 6$) but slightly lower than tropical environments ($9.8 \times 10^{-1} \pm 2.3$) (IAEA, 2010). Crops grown in the control treatment showed about twofold higher TF values as compared with the highest level of waste mica applied treatments (40 g kg^{-1} soil). Between the crops, spinach recorded significantly lower values as compared with lettuce at the second and third cutting stages. Irrespective of soils, graded levels of mica application significantly reduced the transfer of radiocesium from soils to crops. Significant difference was seen in the interaction of soils and treatments; crops and treatments, and crops and soils at the second and third cutting stages, whereas at the first sampling stage, only soils and treatments, and crops and treatments were found to be statistically significant.

Potassium released from waste mica therefore appears to be one of the major ions influencing the radiocesium uptake by plants. Cs/K discriminating factors reported in the literature provide sufficient evidence that K is more efficiently absorbed from soils by different crops (Ciuffo *et al.,* 2003; Kumar *et al*., 2008). Among the soils, Ultisols and Vertisols showed the highest and lowest transfer factors, respectively, and the effect was mainly ascribed to distinct variations with respect to soil pH, clay mineralogy, soil texture and K^+ contents. ^{134}Cs transfer factors to crops were almost identical at the first two sampling stages, whereas in the third cut it was well below the first two sampling stages. At the third sampling stage the high biomass yield might have diluted the Cs content of plants. On the contrary, the potassium concentration was significantly increased from the first to the third cut. This high potassium content leads to a decrease in radiocesium activity in plant parts. Relatively, the low transfer factor in spinach can be attributed to its high exhaustive capacity and high biomass production coupled with high potassium concentration in the leaf tissues (Tang *et al*., 2003; White *et al*., 2003).

Potassium uptake by crops grown in Vertisols was highest under waste mica applied treatments. The effect was ascribed due to enhanced availability of potassium ions through the activity of roots and also to high native soil potassium supplying capacity. The results suggest that treatments enhancing potassium uptake by plants reduce their Cs uptake capacity when both K and Cs are present in the same solution. This is in agreement with the Barber-Cushman mechanistic model which showed that nutrient adsorption mechanisms positively selected potassium over radiocesium, and the sensitivity analysis proved that any parameter modifying potassium uptake will also affect Cs and *vice versa* (Schuller *et al*., 2004; Massas *et al*., 2010). The ability of waste mica to reduce ^{134}Cs uptake by plants increased towards the end of the growing season. This was considered to be due to a slow and continued release of K from mica through the weathering process. Another reason might be due to high fixing capacity of waste mica for cesium (Zachara *et al.,* 2002; Thiry *et al.,* 2005; Sreenivasa Chari, 2010). The data revealed that soil type significantly influenced the ^{134}Cs transfer factors. Ultisols showed nearly 2.3 to 2.6 times higher transfer factors as compared with Inceptisols and Vertisols. The effect was attributed to low pH and dominance of low-charge clay minerals (kaolinites) which have less supportive sites for cesium (Wasserman *et al.,* 2008). This clearly shows the role of clay minerals present in the soil and dominance of the 2:1 type of minerals (smectites, vermiculites and mica) as they play a major role in restricting the transfer of radiocesium from Vertisols and Inceptisols to crops.

4. Conclusion

Application of waste mica to soils had a significant effect on biomass yield, K content and potassium uptake in crops. Graded levels of waste mica reduced the transfer factors of radiocesium: the effect was more pronounced in Vertisols and Inceptisols (nearly 75% reduction) than Ultisols. As compared with control, waste mica at 10, 20 and 40 g kg^{-1} reduced the corresponding ^{134}Cs transfer factors by 52.4, 61.5 and 66.7%, respectively, without any reduction in biomass yield of crops. The results from the present experiment proved that waste mica can be used as a potential amendment to restrict the transport of ^{134}Cs from soils to crops, because of its duel functions: (i) as a sorbent for radiocesium, and (ii) providing slow K supply in soils which excludes cesium from root absorption.

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