Transport of Microplastics in Agricultural Soils - Analyzing Surface Water Runoff as an Environmental Pathway

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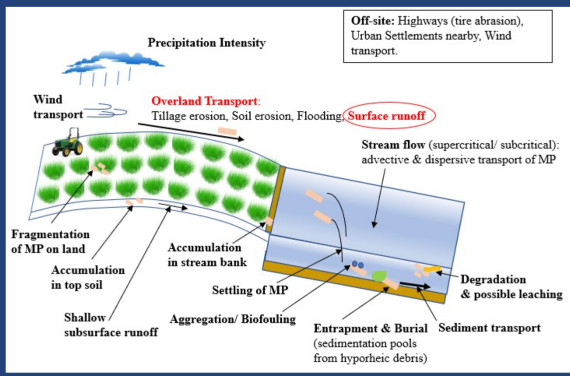

Biodegradable polylactic acid (PLA) microplastic particles (250 - 300 µm) showed a lack of preferential erosion from agricultural topsoil under simulated heavy rainfall on a plot scale.

BACKGROUND

- Current literature highlights limited quantification regarding rainfall induced erosional and transport patterns of microplastics (MP) from agricultural topsoil.
- The aim of this study are: (1) develop a simple, cost-effective technique to detect and quantify luminescent polylactic acid (PLA) particles without extraction from soil media, and (2) analyse PLA MPs transport in agricultural soils under simulated rainfall events in a fallow and crusted plot.

METHODS

- **PLA particles of 250 - 300 µm** were validated under a microscope and proofed for normal distribution using QQ plots.
- **Data Training Set** - To identify parameters for dark room photography and digital camera settings (1200 pictures).
- **Method Validation** - Sets of known values of MP particles were mixed with 10 g soil and photographed in the dark room to ground truth particle counts.
- **Method Evaluation** - PLA particles were added in 7 concentration gradients (0.01%, 0.04%, 0.07%, 0.1%, 0.4%, 0.7%, 1% w/w) with 10 g dry soil and recovery was analyzed (triplicates were used).
- **Field Study** - 3 plots of 1m*1m were prepared and PLA particles were mixed homogenously in top 5 cm.
- **4 g m^{-2}** PLA particles were added only on Day 1 uniformly spread on to top 3 cm of each plot. Due to known properties, this corresponds to **25.2·10^4 particles** in each plot.
- Rainfall simulation (RS) was conducted at an intensity of **59.7 ± 4.25 mm h^{-1}**. Two cycles of RS were conducted per plot – dry and wet run (30 mins each) with a gap of 15 mins
- Two scenarios were tested - Fallow plot (Day 1) and Crusted plot (Day 7)

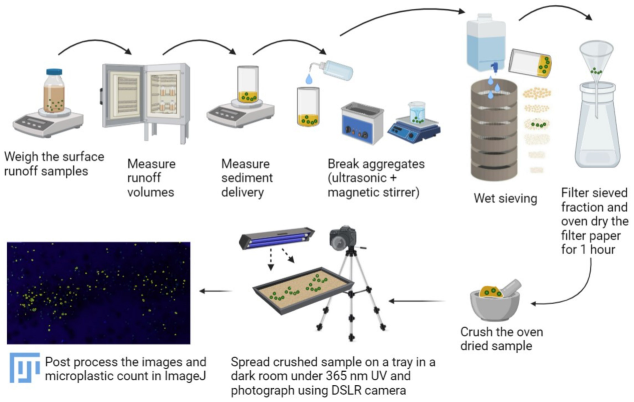

Figure 1: Microplastic detection methodology. **Developed protocol has an efficiency of 89 %**

RESULTS

Figure 2: Photos from field work in Risuty catchment, Czechia

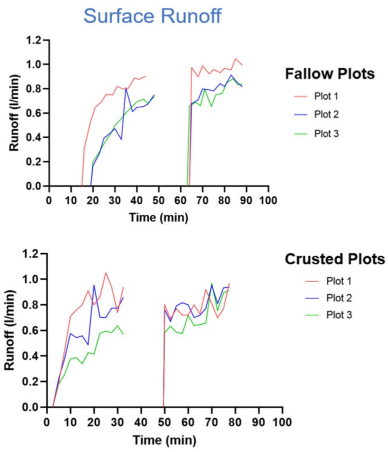

Figure 3: Runoff coefficients were **0.41 ± 0.13 and 0.53 ± 0.2 (n= 6)** for dry and wet runs respectively

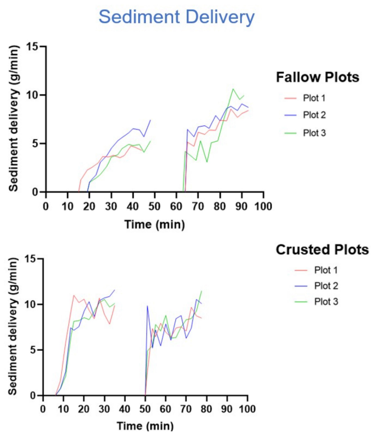

Figure 4: **SD increased by a factor of 2.3** for dry runs between fallow and crusted plots

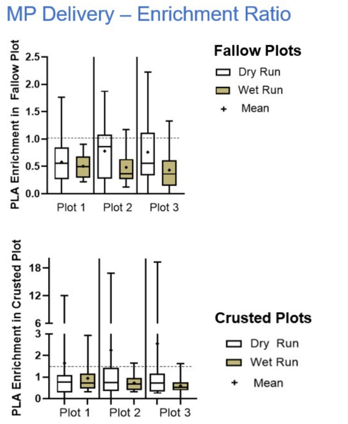

Figure 5: Mean ER for all fallow and crusted plots are 0.095 ± 0.06 and **0.21 ± 0.11** respectively (n=6)

CONCLUSION

- Cheap, convenient, and reliable protocol – applicable for a mix of heavy and low-density fluorescent polymers
- Under naturally relevant input concentration of PLA in a square meter plot only 0.04% of MP was mobilized by surface runoff
- Vertical migration of PLA was observed till a depth of 10 - 12 cm after Day 7, from crusted plots
- Comparison of bio and non-bio MP polymers transport based on density, size, shape should be further investigated.

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Outstanding Student & PhD candidate Presentation contest

Transport of Polylactic Acid (PLA) Microplastics From Agricultural Soil Under Simulated Heavy Rainfall

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Find the abstract here

Objective:

Develop a methodology to identify, extract and quantify the fluorescent PLA MP

Analyze PLA MP transport process under simulated rainfall (field plot scale experiment)

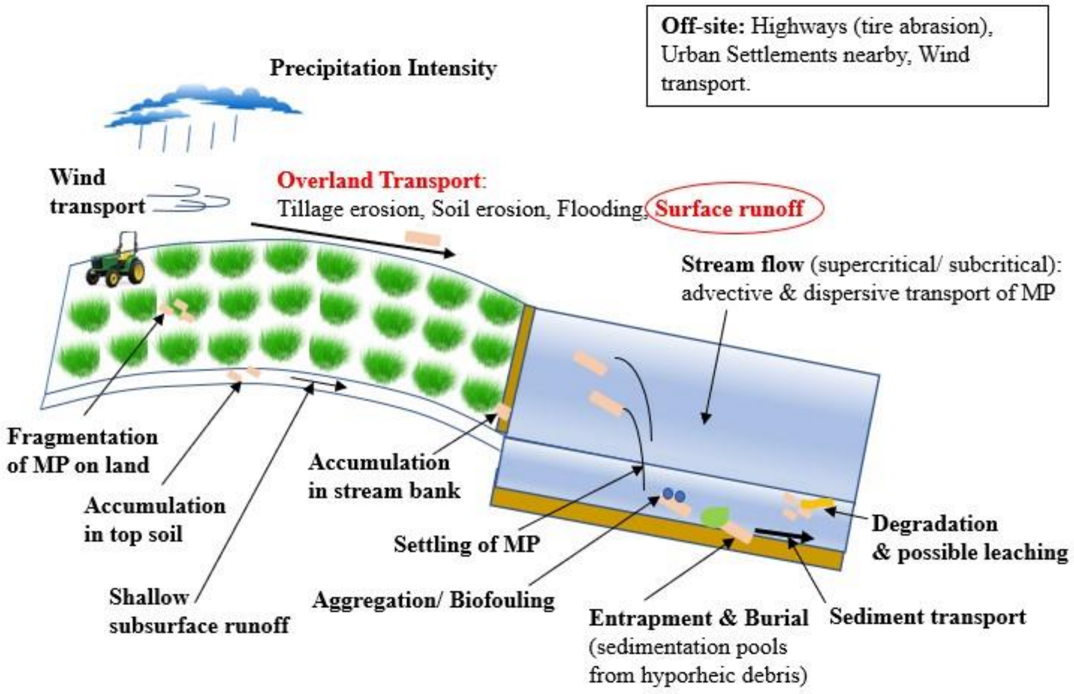
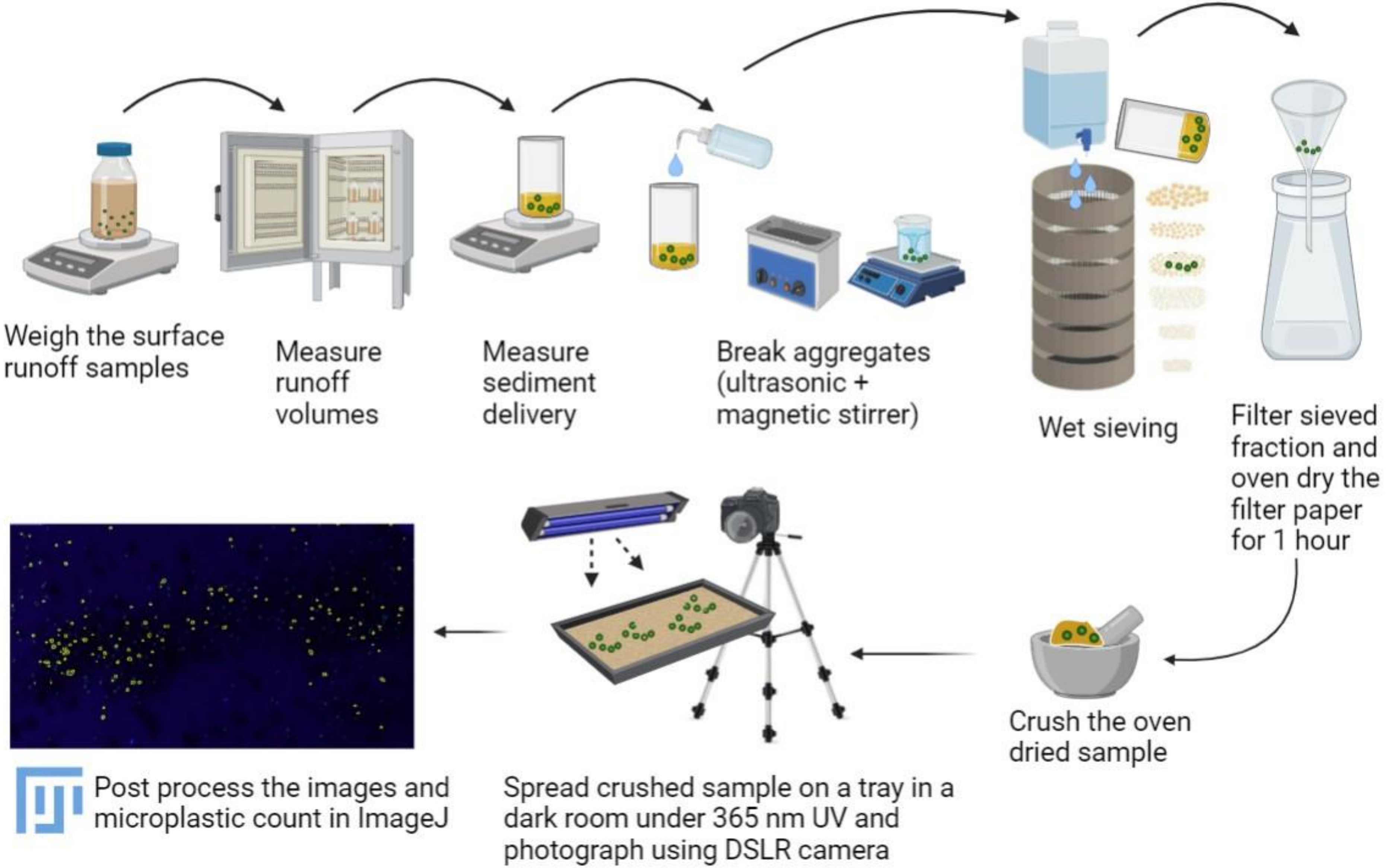


Figure 1: Schematic diagram showing various transport pathways of microplastics from soil to water

Developed Recovery Method for Fluorescent PLA Microplastics



How did I establish the recovery rate of developed methodology?

Determine and **validate PLA MP size distribution**

Data training set – Explore initial conditions for dark room photography

Data validation – Ground truthing camera images from dark room and establish Image J post processing parameters

Establish final conditions for dark room photography and Image J parameters

Data test set on PLA samples added w/w and revalidate precision and recall of the developed process

Advantages of the developed protocol

- Cheap, reliable method with validated reliability of approximately 83%
- Nondestructive method – using fluorescent microplastic as a tracer – helps in future studies related to understanding and analyzing microplastic transport mechanisms in topsoil
- Uses dark room photography under UV light – needs no separation from the soil/ sediment matrix

Field Experiment

Objective: To analyze the transport of PLA particles in agricultural topsoil under simulated heavy rainfall

Study area: Experimental farmland in Czechia (Risuty)

Properties	
Soil Classification	Silty loam
pH	7.61
Bulk Density	1.24 ± 0.04 g cm^{-3}
Porosity	45.5 ± 0.52 %
Soil Moisture	20 % w/w
Organic Carbon	1.22%

Location of Risuty experimental field

Risuty birds-eye view

Preparation of field plots

- **3 plots** were prepared of size 1m*1m
- **2 scenarios** were tested – Fallow plot (Day 1) and Crusted Plot (Day 8)
- All plots were prepared on **Day 1** as:
  - Removal of vegetation cover
  - Ploughing using electric garden hoe (depth of 10-15 cm)
  - Surface application of MP in 0.5*0.5 m grids (4) using a hand-held sieve
  - Ploughing with a hand-held garden hoe (depth of 5 cm approximately)
  - Topsoil compressed by a 30kg lawn roller
  - Slope was kept constant at 10 degrees
- Between days 1 and 8, plots were covered using a tin cover to prevent natural rain, wind, and potential runoff

Rainfall simulation plot preparation and experiment

MP input concentration

- **4 g m^{-2}** MP was added only on Day 1 within the top 3 to 4 cm
- Due to known properties, this corresponds to **25.2· 10^{4} particles** in each plot
- Topsoil MP concentration was collected from 6 random point locations (as a composite sample of app. 30g) from <1cm layer for **calculating background/ mean MP concentration**
- **Enrichment ratio (ER)** was calculated as mean MP concentration in delivered sediments/ mean MP concentration in topsoil (ER <1 indicate depletion of MP in delivered sediment and vice versa)
- A relatively low input concentration was chosen intentionally to be more realistic to natural conditions – analyzing transport as a snapshot in time

Rainfall simulation and sampling parameters

- Rainfall simulation had a mean drop diameter of 1.8 mm, with a mean drop velocity of 6.7 ± 0.45 m s^{-1}. RS was calibrated to near constant rainfall intensity of 59.7 ± 4.25 mm h^{-1}, using a plastic cover over 1 m* 1m (mean coefficient of variation for 3 simulations = 6.24%)

- 2 cycles of RS were conducted per plot – dry and wet run (30 mins each) with a gap of 15 mins

- From the visual observation of surface runoff start, samples were collected in glass jars every 2.5 mins

- Soil moisture of topsoil was measured before and after (within 10 mins) of each run (dry and wet) at 12 locations from within the plots

- At end of crusted plot RS (Day 8) – vertical samples were taken from within the plots using a hand-held auger (4 cm dia.) as composite layers of 0 – 5, 5 – 10, 10 – 15 cm

End of simulation on Day 1

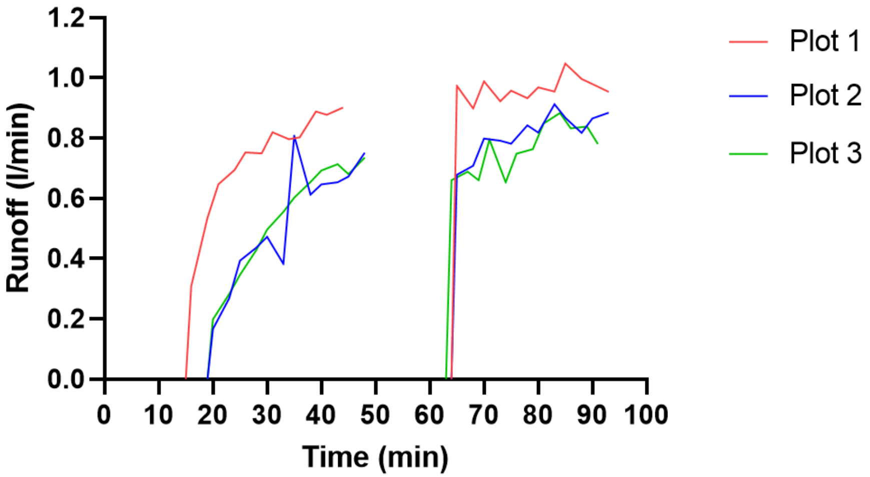
Crusted plot on Day 8

Vertical Sampling locations after crusted plot RS

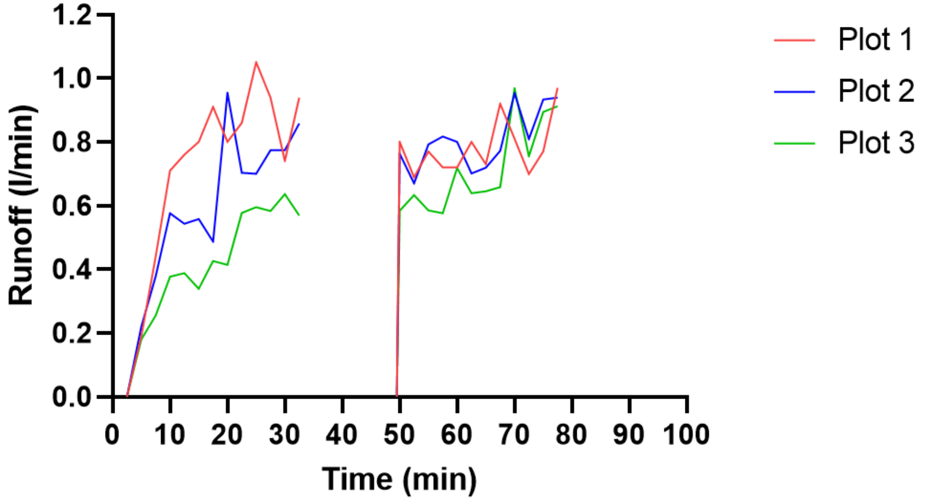
Vertical sampling using an auger

Results – Surface runoff

Fallow Plots



Crusted Plots

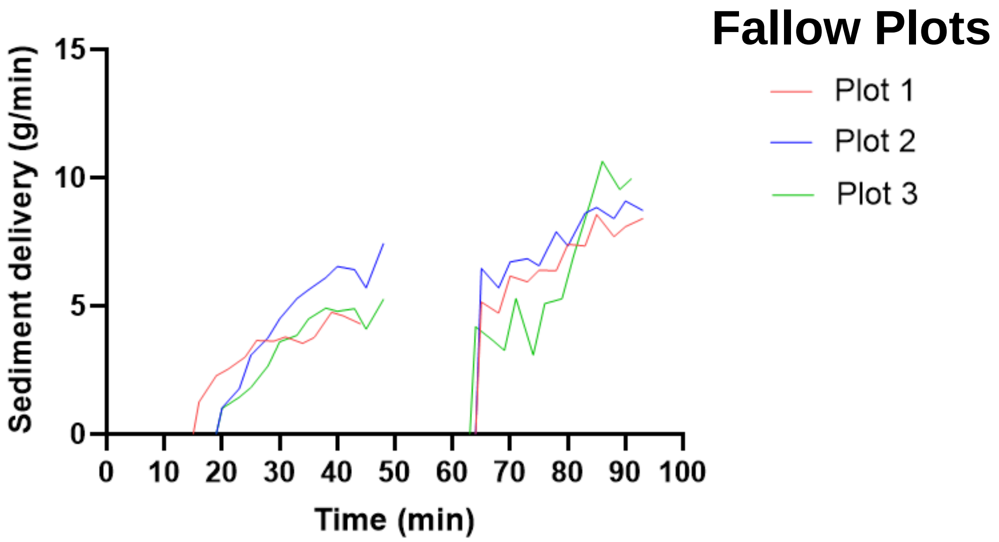


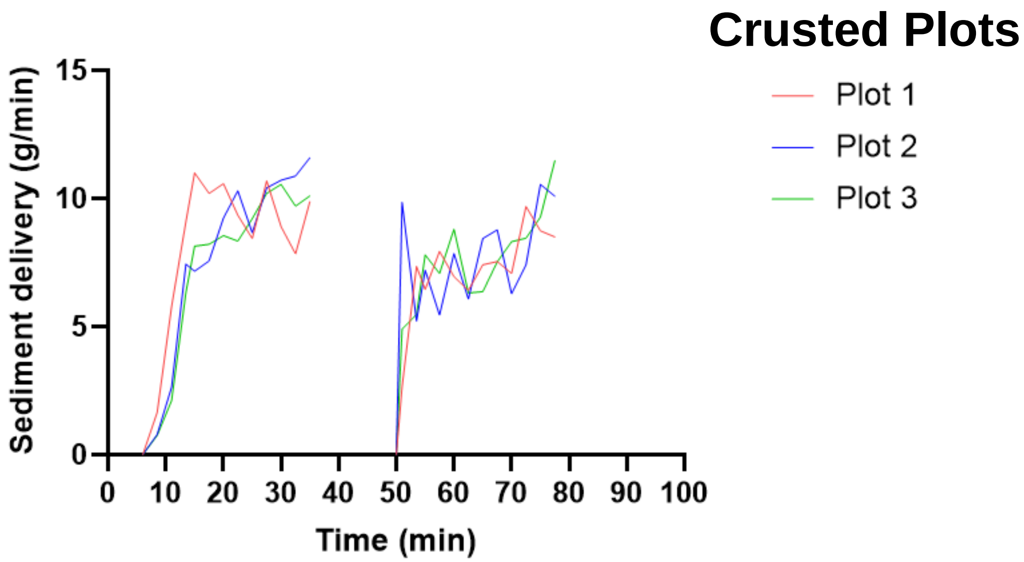
- Runoff coefficients of 0.41 ± 0.13 and 0.53 ± 0.2 (n= 6) for dry and wet runs respectively
- Runoff is variable for dry runs in the crusted plots due to variable initial soil moisture conditions
- Wet runs produced similar runoff rates due to similar SM conditions after dry runs
- Runoff volumes similar for fallow and crusted plots

Scenario	Plots	Before RS (vol.-%)	After dry run (vol.-%)
Fallow	1	8.45 ± 1.03	31.13 ± 5.1
	2	7.64 ± 0.76	36.2 ± 3.01
	3	7.95 ± 0.86	35.33 ± 4.2
Crusted	1	11.79 ± 1.63	28.28 ± 4.22
	2	13.56 ± 2.86	26.55 ± 3.81
	3	14.19 ± 1.99	32.52 ± 2.15

Mean SM as vol.-% from 12 plot locations (topsoil <6cm), variability is indicated as ± SD

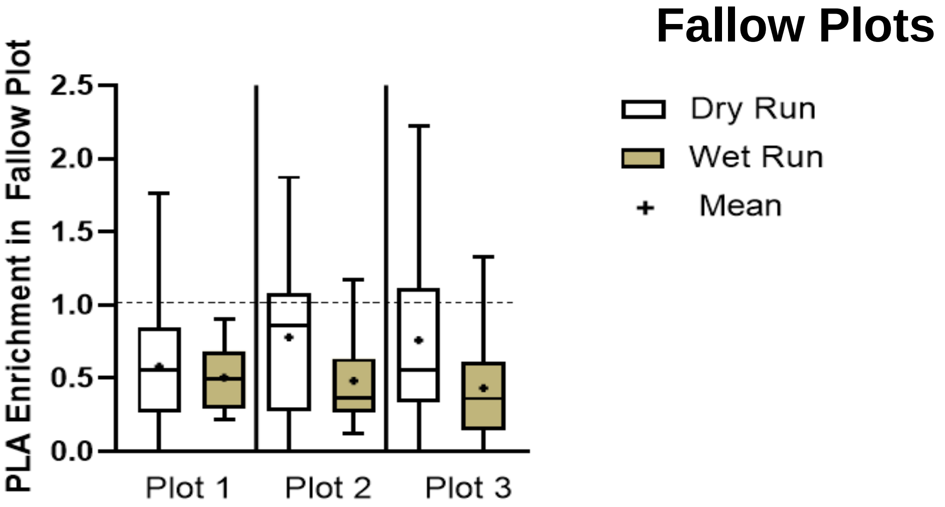
Results – Sediment delivery

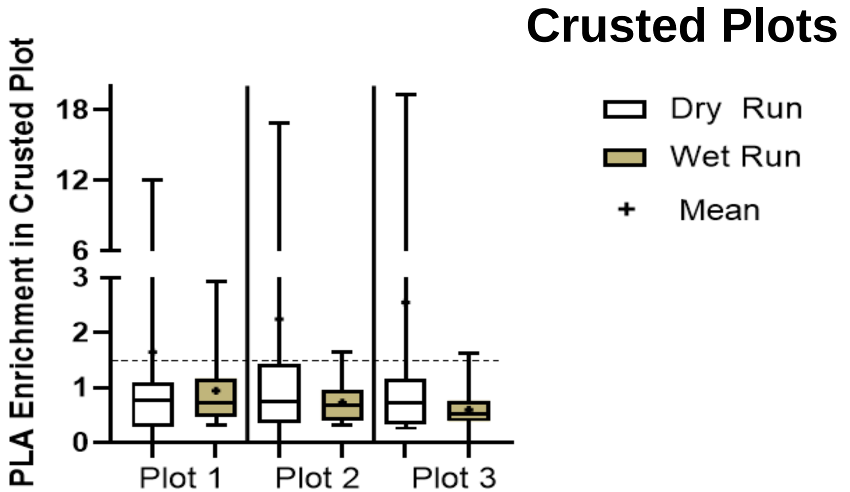




- Sediment delivery (SD) rates of fallow and crusted plots, in general, have similar dynamics to surface runoff
- SD increased by a factor of 2.3 for dry runs between fallow and crusted plots (mean SD increase for dry runs, but similar for wet runs)

Results – MP delivery and transport



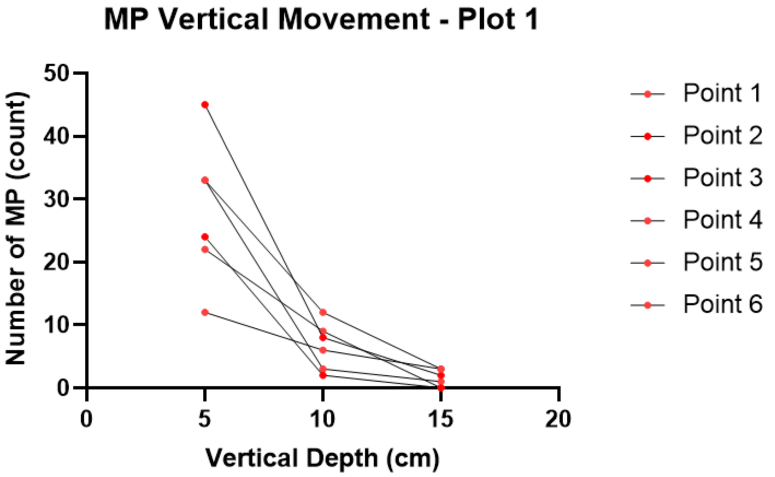


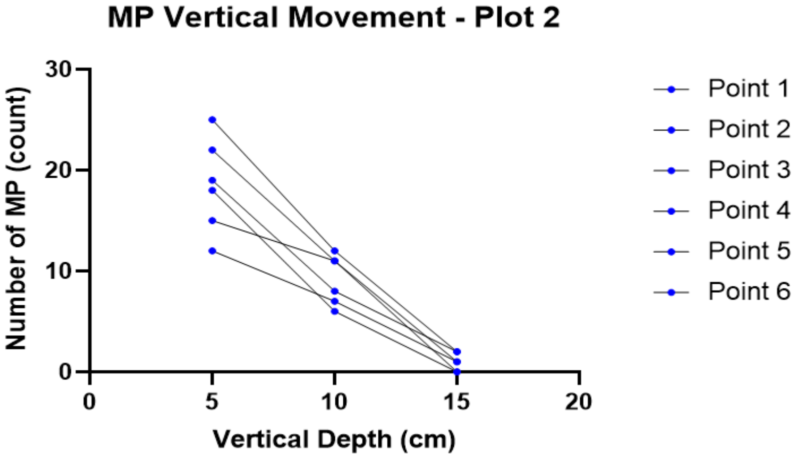
- We observe a **depletion of PLA in the sediment** (ER <1 = lower concentration of microplastics as compared to start/ background topsoil concentration)

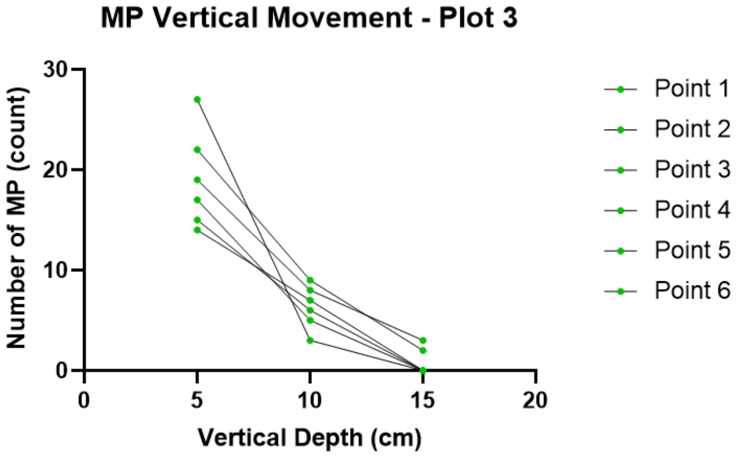
- Mean ER for all fallow and crusted plots are 0.095 ± 0.06 and **0.21 ± 0.11** respectively (n=6)

- Significant increase in ER for dry runs amongst fallow and crusted (0.0812 ± 0.05 vs **0.228 ± 0.14**; n=3) as compared to wet runs (0.11 ± 0.06 vs 0.17 ± 0.07; n=3)

Results – MP movement in vertical direction







- No degradation was observed in PLA particles over a period of 7 days (mean difference in weight 1.3 ± 1.08%; n=10)

Takeaways of field experiment

- Known concentrations of microplastic were inputted and their transport was analyzed as a snapshot in time through controlled rainfall simulations over fallow (newly tilled) and crusted (no-till) plots within a period of 7 days

- Crusting of plots showed increased sediment delivery rates and in turn, higher mean delivery rates of microplastics

- Although, in general, we observe a depletion of PLA in the runoff from the plots

Summary

- Cheap, convenient, and reliable protocol – applicable for a mix of heavy and low-density fluorescent polymers
- PLA is effective as a cheap tracer to analyze the transport of bio-polymers (compare with PE movement – upcoming joint fieldwork in Prague)
- Transport wise PLA showed an ER < 1 (depletion) and we also observed loss of PLA below the input layer (0-5 cm)
- Crusting of plots (1 wet-dry cycle) increased horizontal movement of PLA
- Does input concentration of MP plays a role in its transport – is there a threshold to MP movement?
- How significant are the transport mechanisms amongst bio and non-bio polymers?

Thank you!

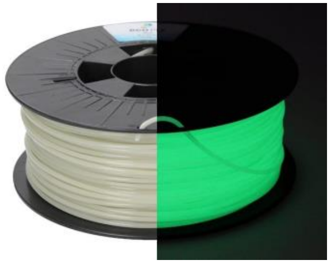
This project has received funding from European Union’s Horizon 2020 research and innovation program under the Marie Skłodowska-Curie grant agreement No 955334.

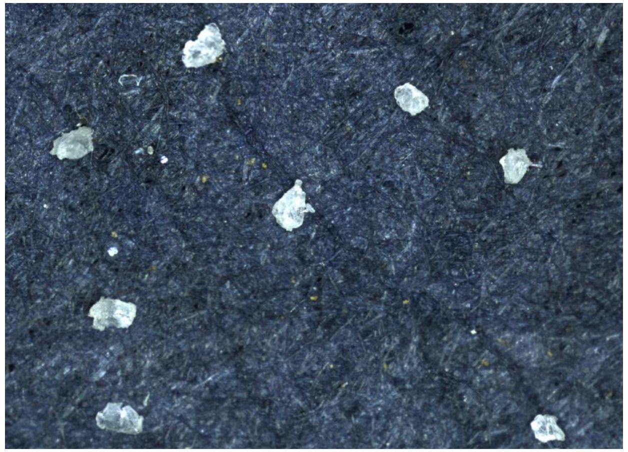
Additional Material: Method Validation Details

- Known number of particles were put under a microscope and camera (in a dark room) to analyze the particle size and pixel area respectively

- Size and area distribution within 250 – 300 µm was validated using a microscope and a camera and proofed for normal distribution

- Through photography the mean area of particles in terms of pixel size was 23.92 ± 0.71 (SD) with a minimum of 5 and a maximum of 56 pixels covered





Properties	PLA
Density	1.24 g/cm³
Melting Point	160 °C
Color	Grey (Luminescent green)
Shape	Particles (Heterogenous)
Size	250 – 300 µm
Production	Dry milling and sieving

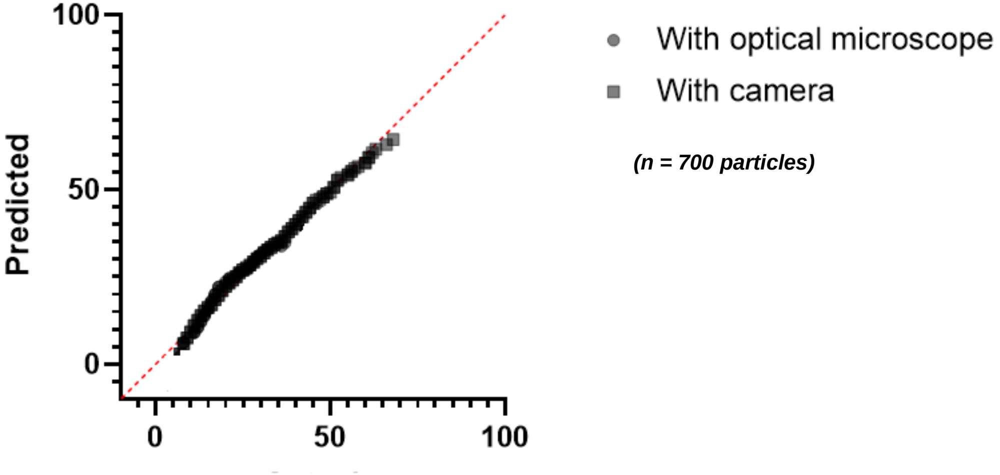


Figure 2: Size distribution of PLA microplastic fraction subsample determined under stereomicroscope and camera and proofed for normal distribution using QQ plot

Data Training Set - Exploring conditions

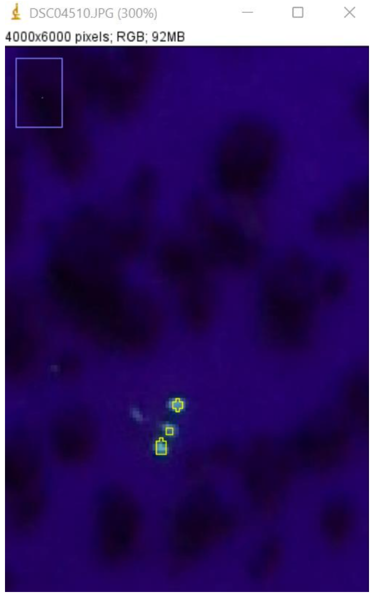
Method development

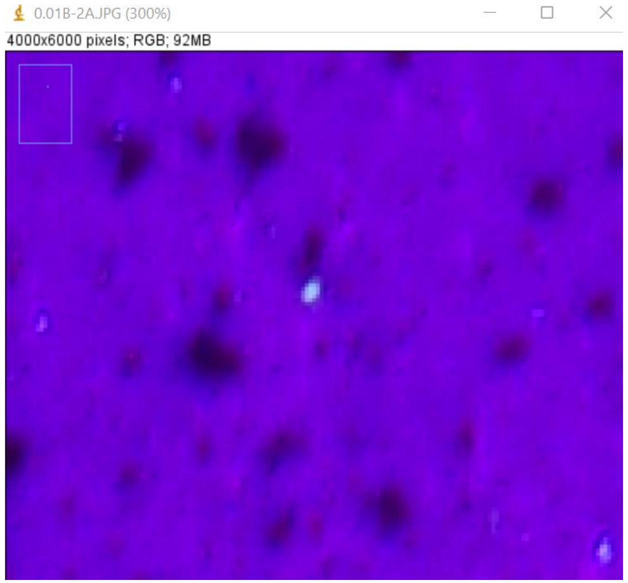
- Testing of digital cameras – two different models of Sony alpha models – both yielded the same results
- Dark room conditions – app 12m^2, all potential light sources were blocked, only operator was allowed, a tripod stand was built separately to hold the camera and reduce mechanical vibration during shutter, tripod stand was covered via cardboard box
- Various combinations of ISO (100, 200, 400, 800, 1600, 3200) and exposures (1/13, 2, 5, 15, 30) were tested at different times of day - morning 10 am, noon (12 – 1 pm), evening (5-6 pm)
- Two different sources of UV light were tried – 275 nm, and 350 nm, the combination of both
- Darkroom photographs were taken to correct for dead pixels within the camera lens
- These images were analyzed via Image J to check for the best photographic settings

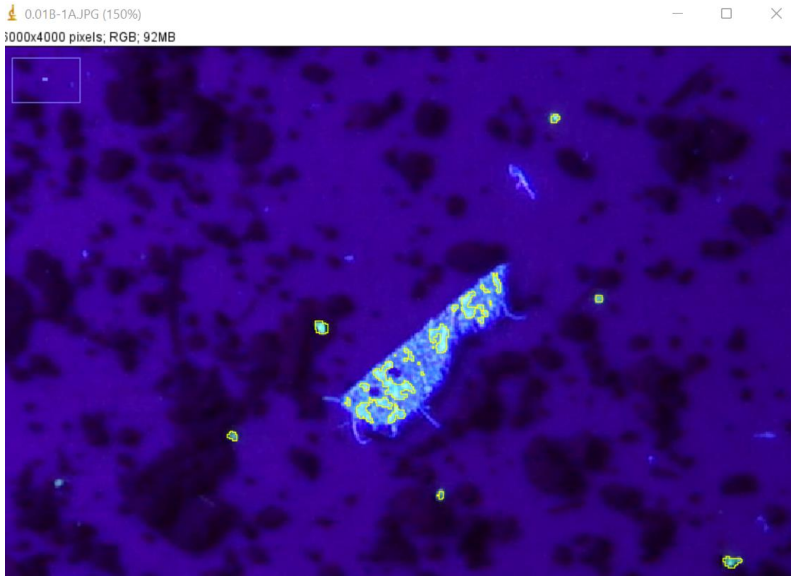
Data Validation – Ground truthing camera images

Method optimization and ground truthing

- Sets of known values of MP particles were mixed with 10 g soil and photographed in a dark room to ground truth particle counts
- Actual (ground truth) versus Predicted (results) class = **TP** – actual MP, **FN** – MP but the camera did not detect it correctly, **FP** – not MP but the camera says it as MP (quartz/ synthetics/ reflections from OM), ~~**TN** – all other particles than MP~~
- Precision, Recall and F score – To determine the accuracy of results versus ground truth images – used to tweak Image J parameters



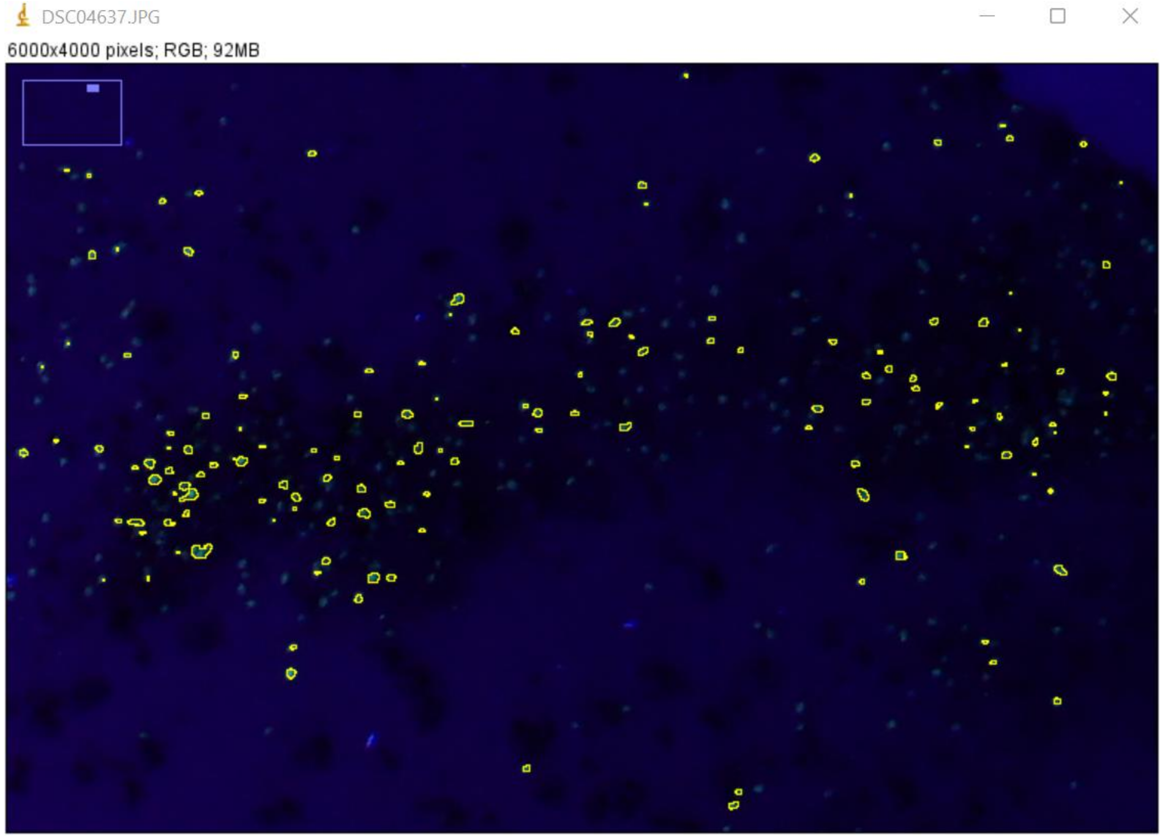




After Recovery

No. of MP	TP	FN	FP	Recall	Precision	F-score
5	2.00	0.00	3.00	1.00	0.40	0.57
10	7.00	1.00	3.00	0.88	0.70	0.78
15	13.00	3.00	2.00	0.81	0.87	0.84
20	17.00	5.00	3.00	0.77	0.85	0.81
30	23.00	10.00	7.00	0.70	0.77	0.73
40	36.00	2.00	4.00	0.95	0.90	0.92
50	40.00	15.00	10.00	0.73	0.80	0.76
60	53.00	2.00	7.00	0.96	0.88	0.92
80	71.00	5.00	9.00	0.93	0.89	0.91
100	89.00	12.00	11.00	0.88	0.89	0.89
150	132.00	22.00	18.00	0.86	0.88	0.87
200	172.00	12.00	28.00	0.93	0.86	0.90

Recall: 0.86 ± 0.09
Precision: 0.82 ± 0.09
F Score: 0.83 ± 0.07



Clustered microplastic particles were calculated as the ratio of cluster pixel area/ mean particle pixel area (24 pixels)

Final conditions for dark room photography

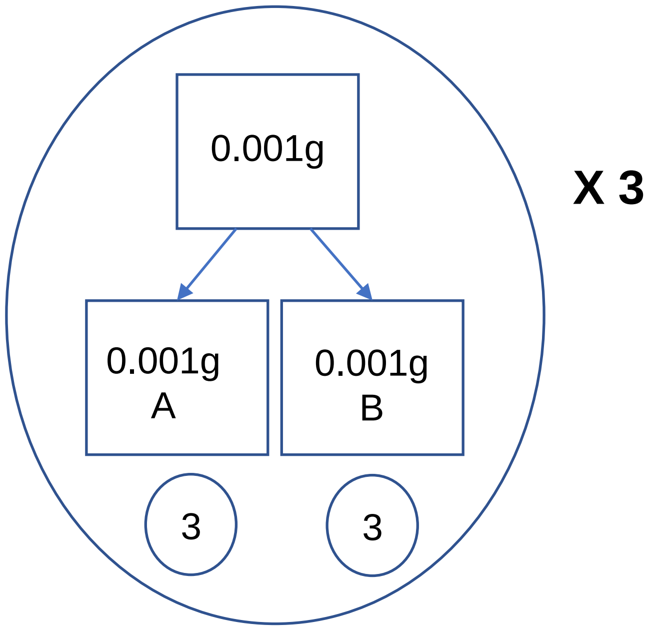
Uniform illumination
300 nm wavelength LED – 2 mins
RI of PLA = 1.49
350 nm UV

Sony a6500
F 5.6
ISO 100
Exposure 2 sec
Full frame pictures

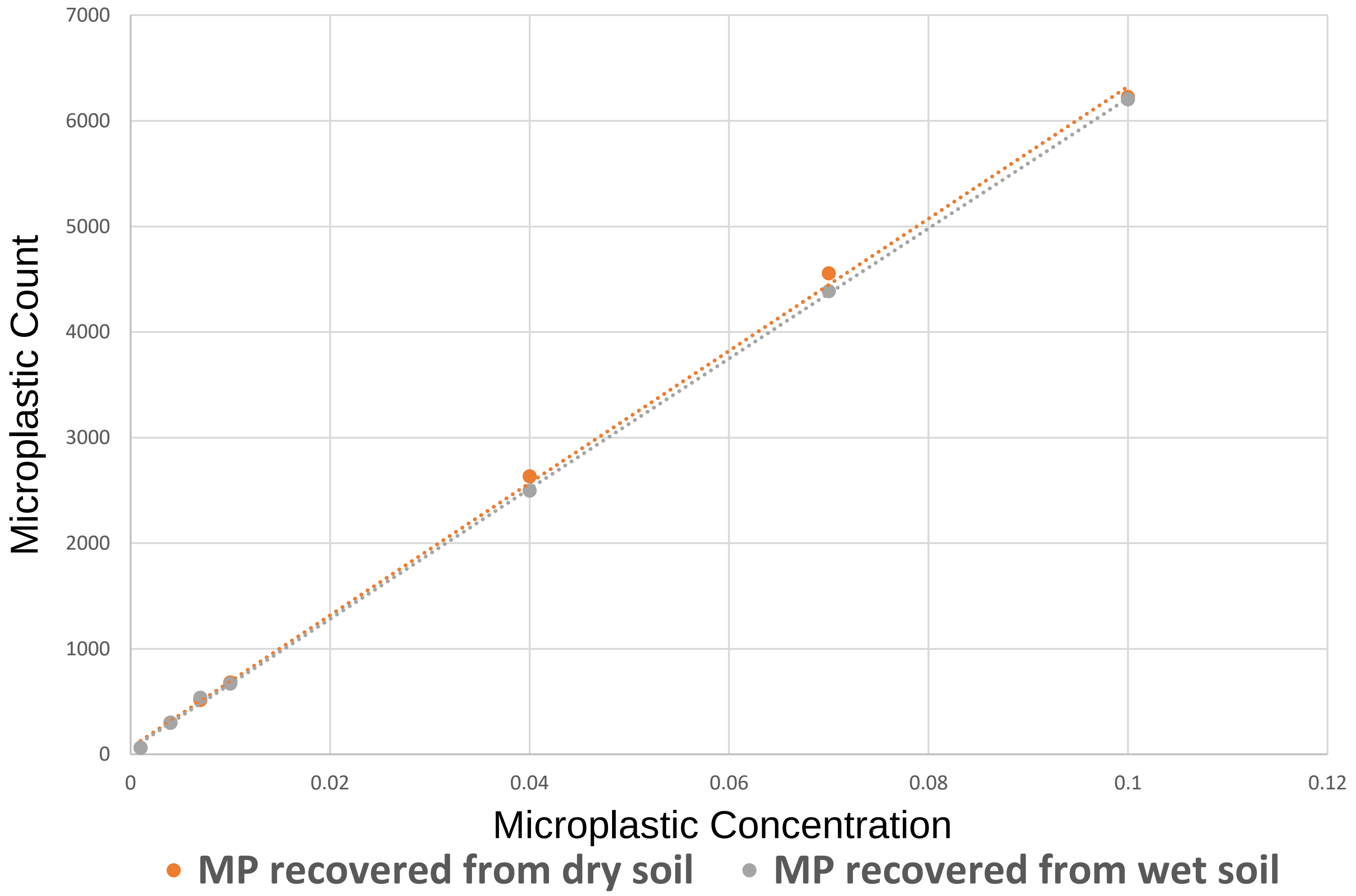
Image calibrated as per scale
Threshold application (Hue: 20 – 145, Sat: 0-250, B: 40 – 250)
Watershed application
Particle count analysis

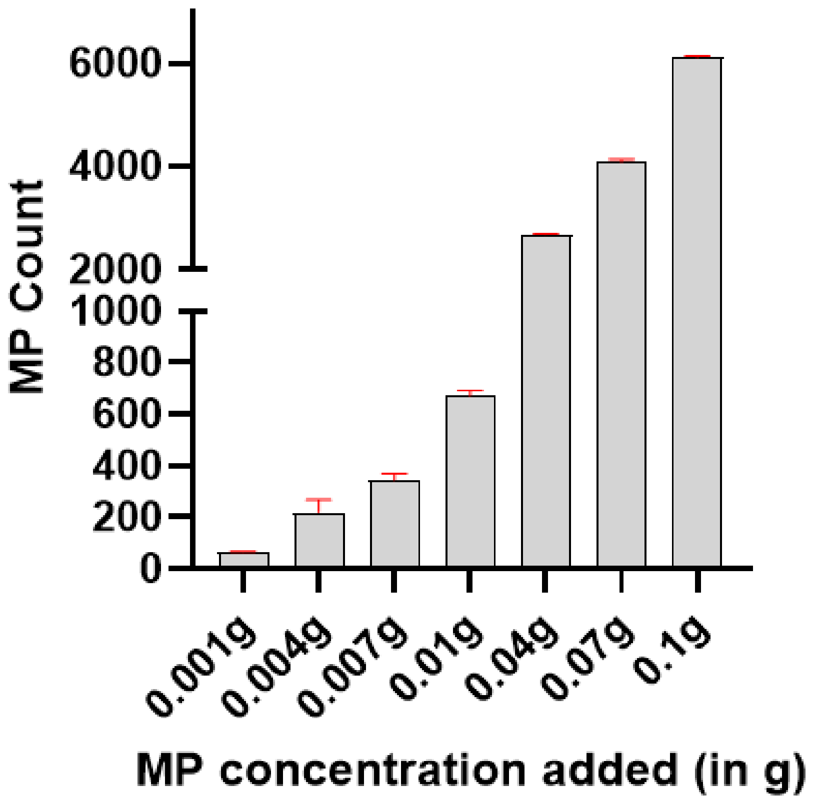
Method evaluation

- 10 g Silty loam (16% sand, 59% silt, 25% clay) air dried and sieved at 2mm – taken from Risuty field
- PLA added at 7 concentration gradients - 0.01%, 0.04%, 0.07%, 0.1%, 0.4%, 0.7%, 1% w/w
- Three triplicates for each sample
- Each triplicate is divided into two subsamples (A and B) for photography in the darkroom
- Each subsample was photographed thrice to observe the operator-made variance in the distribution (18 dry and 3 wet, n =21)
- Photographs are taken for:
    - dry soil + PLA mixture
    - then mixed with 500 ml distilled water and incubated at 4 °C for 7 days
    - Sieved and oven dried – photo taken again
    - Compare the number of particles in dry versus wet conditions



Data Test Set- Results





Equation	R^2
$y = 50614x + 115.99$	0.99
$y = 62615x + 63.423$	0.99