Supplementary information

for

Emerging investigator series: Suspended air nanobubbles in water can shuttle polystyrene nanoplastics to air-water interface

Kenneth Mensah[a], Sudheera Yaparatne[a], Andre L. Magdaleno[b], Sergi Garcia-Segura[b], Onur G. Apul*[a]

[a] Department of Civil and Environmental Engineering, University of Maine, Orono, ME 04469, United States

[b] School of Sustainable Engineering and the Built Environment, Arizona State University, Tempe, AZ 85287, United States

Corresponding author: email: onur.apul@maine.edu; Phone: +1 207.581.2981 Fax: 207.581.3888

Text S1

Materials and reagents

100 nm polystyrene (PS) latex indicator standard (0.01% weight/volume) was purchased from Malvern Panalytical and used as a model nanoplastic. PS is the most utilized plastic polymer for plastic pollution and toxicity studies.[1–35] Sodium hydroxide (analytical grade, 99%) and hydrochloric acid (ACS reagent grade, 37%) were procured from Acros Organics.

Nanobubble generation

Air nanobubbles were generated in a 32-gallon nanopure water (NW) using a Moleaer XTB 25 generator. The system was made up of a gas pressure of 100 PSIG, a gas flow rate of 1 LPM, and 13 PSI water pumping pressure for 1 h. The generated nanobubbles were transferred to 1 L polypropylene bottles, ensuring it was filled to the brim with no headspace, and then stored in a refrigerator at 4 °C. The number concentration and size distribution of the bubble were analyzed using NanoSight NS 300.

Text S2

UV-vis spectroscopy

The concentration of nanoplastics was analyzed using a Hach DR 6000 UV-vis spectrophotometer in a 1 cm plastic cuvette at a wavelength of 249 nm. To ensure data reliability in the measurements, each experiment was performed in triplicates and each sample was measured at least five times with the UV-vis spectrophotometer, and the average of three close values was selected. The concentrations of nanoplastics were determined using a linear equation generated from the calibration curve (Fig. S1) that relates the absorbance to concentration. The calibration curve in Fig. S1 shows that nanoplastics can be detected in both DI water and nanobubble solution without any interference by the nanobubbles.
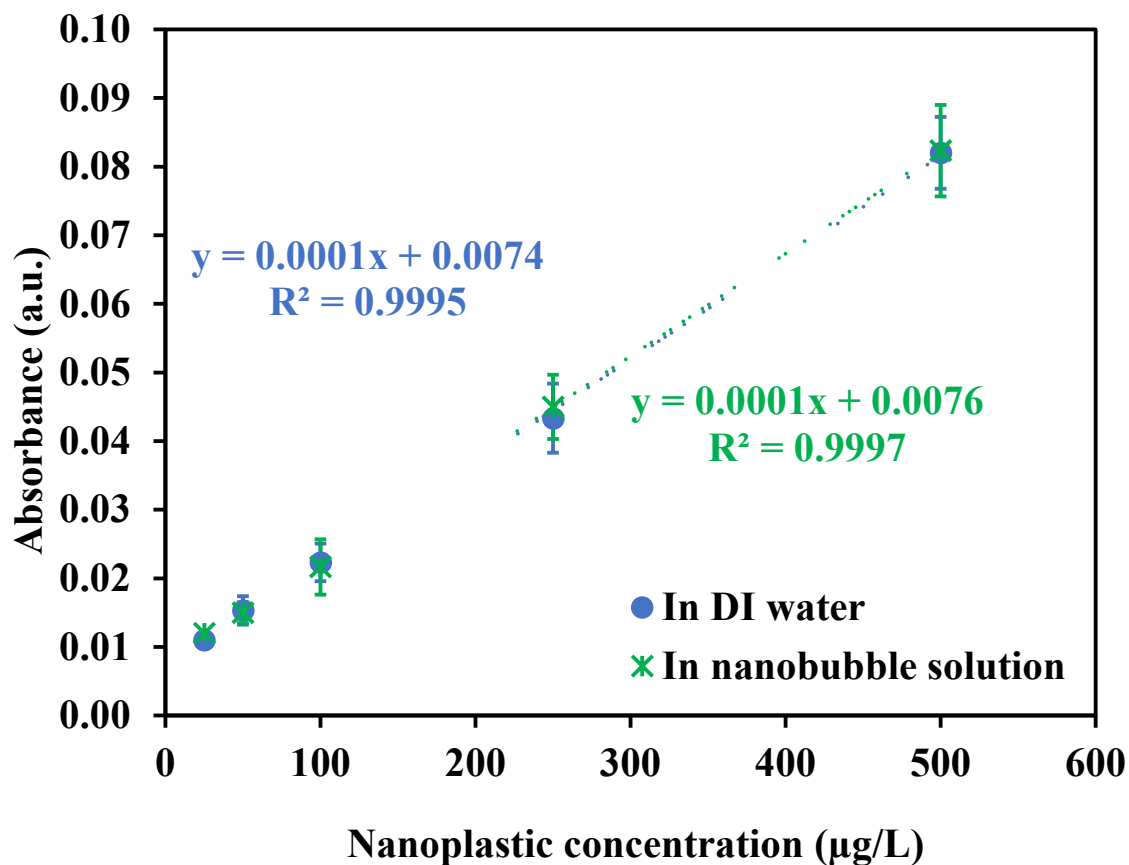

Fig. S1. UV-vis calibration curve for nanoplastics in nanopure and nanobubble water.

Nanoparticle Tracking Analysis (NTA)

NanoSight NS300 (Malvern Panalytical, UK) was employed for the assessment of number concentration, average size, and size distribution of triplicate 1 mL samples of nanoplastics, nanobubbles, and the mixture of nanoplastics and nanobubbles. The instrument characterizes nano-entities (particles and bubbles) based on the nanoparticle tracking analysis (NTA) principle as explained elsewhere.[36,37] Our NanoSight NS300 is equipped with a green laser and a syringe pump. Each measurement entailed 60 seconds of video recording at a camera level of 13-15. The detection threshold was set at 5-20. The NTA software analyzed five consecutive video recordings for each NanoSight measurement, producing a number/size distribution, with the reported results representing the averages and standard error.

Zeta potential

The zeta potential of the nanobubbles and nanoplastics from triplicate measurements was analyzed using Zetasizer Nano-ZS. For each sample, the initial and final pH after the experiment, presented in Table S1 with the standard error (SE), was measured using MultiLab IDS 4010-3W. Prior to the zeta potential measurement, the refractive index and absorption values of nanobubbles and nanoplastics were set as shown in Table S2. The dispersant was set to water with temperature = 25 °C, viscosity = 0.8872 cP, refractive index = 1.330, and dielectric constant = 78.5. Samples were loaded in a DTS1060C-Clear zeta cell and the equilibration time was set to 120 s. The zeta potential was automatically calculated by the Zetasizer Nano-ZS software using the Smoluchowski model and an F(Ka) value of 1.5. As a control measurement, the zeta potential of DI water was measured to be -0.75 ± 0.37.
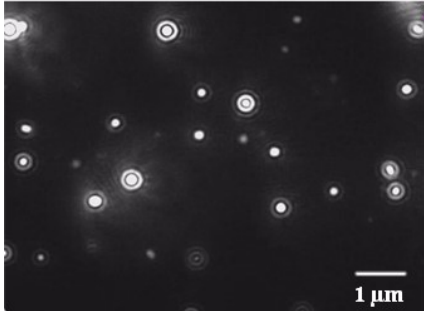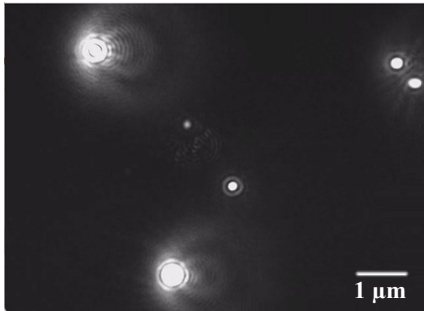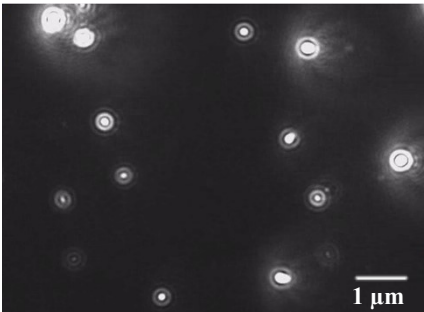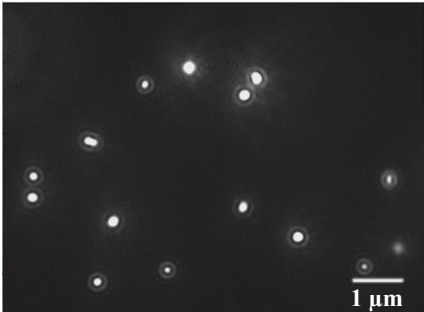
Table S1 Changes in pH before and after stirring
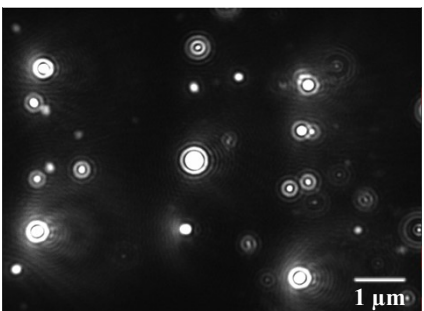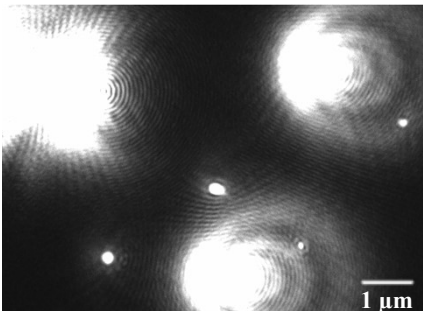
Expected pH	Nanobubbles		Nanoplastics in DI water	
	pH before ± SE	pH after ± SE	pH before ± SE	pH after ± SE
9.0	9.14 ± 0.15	7.32 ± 0.27	9.10 ± 0.04	7.38 ± 0.07
Pristine pH	6.33 ± 0.18	6.27 ± 0.15	6.28 ± 0.22	6.11 ± 0.33
5.0	4.70 ± 0.14	4.56 ± 0.19	5.04 ± 0.17	5.02 ± 0.13
3.0	3.11 ± 0.01	3.09 ± 0.01	3.08 ± 0.01	3.05 ± 0.02

Table S2 Parameters of nanobubbles and nanoplastics used in zeta potential measurement

Sample	Material	Refractive index	Absorption
Nanobubbles	Air	1.00	0.00
Nanoplastics	Polystyrene latex	1.59	0.01
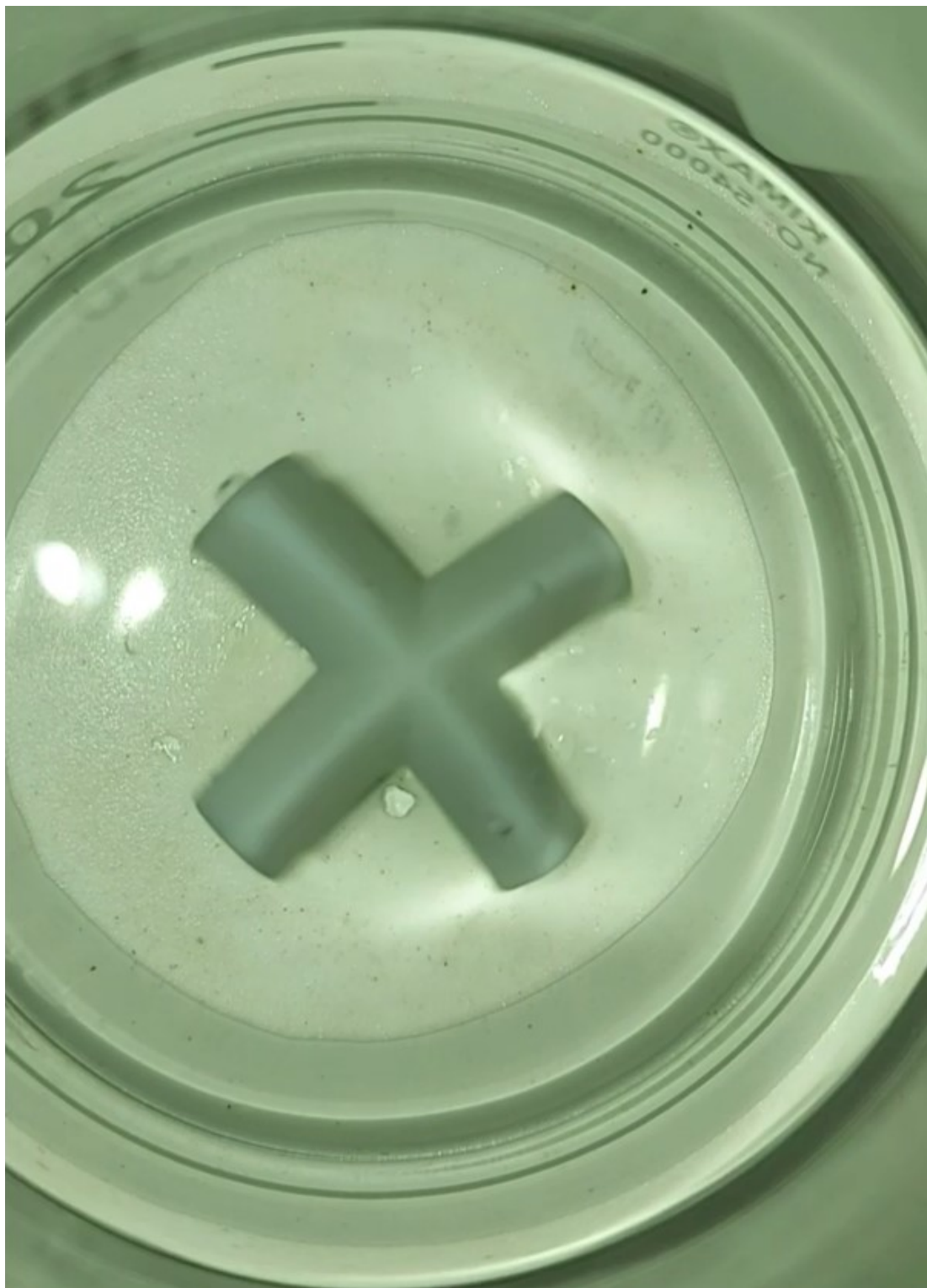
Fig. S2. Visible appearance of a thin film after stirring NPs with nanobubbles at pH 3.

	Before stirring	After stirring
Nanobubbles		
Nanoplastics		
Nanoplastics + Nanobubbles		

Fig. S3. NTA micrograph of nano-entities in the bulk phase before and after stirring at pH 3 for 5 min and 400 RPM.
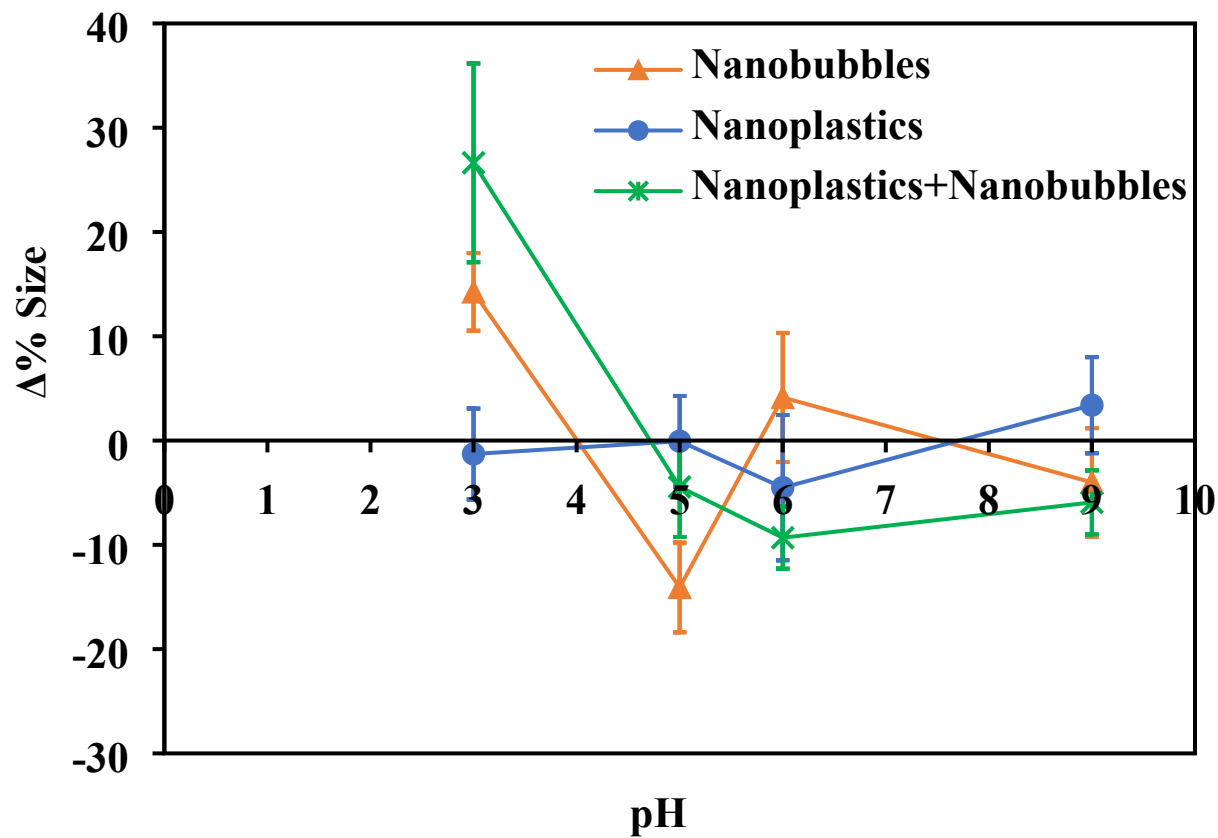
Fig. S4. Changes in particle size of nanobubbles, nanoplastics, and nanoplastics with nanobubbles after 5 min stirring at various pH (Conditions: initial nanoplastic concentration = 38 μg/L, stirring speed = 400 rpm, stirring time = 5 min).

Text S3 Rising and settling calculations

Computations for settling velocity of nanoplastics

The following set of equations from a previous study was used to estimate the settling velocity of nanoplastics.[38,39] Polystyrene latex are nanospheres, hence shape factor = 1, and diameter of the nanoplastic (d) = equivalent sphere diameter

The settling velocity was calculated using Stoke's law in Eq. 1 assuming a laminar flow:

$$v_s = \frac{(\rho_p - \rho_w)d_p^2 g}{18\mu} \tag{1}$$

Where, v_s = settling velocity at laminar flow (m/s)

ρ_p = density of nanoplastic (kg/m³)

ρ_w = density of water at 20 °C (998.2 kg/m³)

d_p = diameter of nanoplastic (m)

g = acceleration due to gravity (9.81 m/s²)

μ = dynamic viscosity of water at 20 °C (1.002 × 10⁻³ kg/m.s).

The validity of the v_s was verified by confirming with the Reynolds number (Re) (Eq. 2) given by:

$$Re = \frac{w d_p \rho_w}{v} \tag{2}$$

Where, v = kinematic viscosity of water at 20 °C (1.003 × 10⁻⁶ m²/s)

w = iterative settling velocity (m/s) (Eq. 3), $w = v_s$ in the first iteration

$$w = \sqrt{\frac{4d_p}{3C_D}\left|\frac{\rho_p - \rho_w}{\rho_w}\right|g} \tag{3}$$

where, C_D = Drag coefficient (Eq. 4)

$$C_D = \frac{24}{Re}, \text{ for } Re < 0.5 \tag{4}$$

Plastic-bubble aggregate rising velocity calculations

For flotation to occur, nanobubbles need to overcome Brownian motion and rise.[40] The critical size required by bubbles (d_b) to overcome Brownian motion and rise can be calculated using Eq. 5.

$$d_b = \left(\frac{216kT\mu}{\pi g^2 (\rho_w - \rho_b)^2 t}\right)^{1/5}$$

(5)

Where, k = Boltzmann's constant (1.38×10^{-23} J/K)

T = absolute temperature (293 K)

t = time (s)

d_b = bubble diameter, m

According to Crittenden et al. (2012), the following expressions can be used to determine the theoretical rising velocity of a particle-bubble floc.[40]

The formula for calculating the density of the plastic–bubble floc (ρ_{pb}, kg/m³) is given by (Eq. 6):

$$\rho_{pb} = \frac{\rho_p d_p^3 + N_b \rho_b d_b^3}{d_p^3 + N_b d_b^3}$$

(6)

Where, ρ_b = air bubble density (kg/m³)

N_b = number of bubbles attached to floc particle

The equivalent spherical diameter of the plastic–bubble aggregate (d_{pd}) was determined using Eq. 7.

$$d_{pd} = (d_p^3 + N_b d_b^3)^{\frac{1}{3}}$$

(7)

The rising velocity of a plastic-bubble aggregate (v_{pb}) is given by (Eq. 8):

$$v_{pb} = \left(\frac{g(\rho_w - \rho_{pb})d_{pb}^{1.75}}{33.75\rho_w^{0.25}\mu^{0.75}} \right)^{1/1.25}$$

(8)

The minimum volume of gas for flotation to occur is expressed by (Eq. 9):

$$\frac{\phi_g}{\phi_p} = \frac{\rho_p - \rho_w}{\rho_w - \rho_b}$$

(9)

Where, ϕ_g = minimum volume of gas needed for flotation (mL/L)

ϕ_p = volume of particles (mL/L)

Text S4 Stirring speed and velocity gradient formulas

The overall plastic-bubble collision rate depends on the rates of macroscale flocculation, microscale flocculation and differential settling flocculation between nanoplastics and nanobubbles.[40] Furthermore, the rate of plastic-bubble attachments (r_{pb}) can be determined by the particle concentrations and a collision frequency function (β_{pb}) as depicted in Eq. 10.

$$r_{pb} = \alpha\beta_{pb}n_p n_b \tag{10}$$

Where, r_{pb} = rate of attachment between nanoplastics and nanoplastics

α = collision efficiency factor (attachments per collision). $\alpha = 1$, assuming effective destabilization of nanoplastics and nanoplastics at pH 3.

β_{pb} = overall collision frequency between nanoplastics and nanoplastics (m^3/s) (Eq. 11)

n_p = nanoplastics concentration

n_b = nanoplastics concentration

$$\beta_{pb} = \beta_M + \beta_\mu + \beta_{DS} \tag{11}$$

β_M = macroscale collision frequency, $= \dfrac{1}{6}\overline{G}(d_p + d_b)^3$ (12)

β_μ = microscale collision frequency, $= \left(\dfrac{2kt}{3\mu}\right)\left(\dfrac{1}{d_p} + \dfrac{1}{d_b}\right)(d_p + d_b)$ (13)

β_{DS} = differential settling collision frequency, $= \dfrac{\pi(\rho_p - \rho_w)g}{72\mu}[(d_p + d_b)^3(d_p - d_b)]$ (14)

where, $\overline{G}$ = RMS velocity gradient (s^{-1}) (Eq. 15)

$$G = \sqrt{\dfrac{P}{\mu V}} \tag{15}$$

Where, P = power input of horizontal mixing (W = kg·m²/s³) (Eq. 16)

V = volume of nanoplastic-nanobubble suspension, m^3

$$P = \dfrac{C_{DP}A_p\rho v_R^3}{2} \tag{16}$$

Where, C_{DP} = drag coefficient on paddle (Eq. 17)

A_p = projected area of paddle (m²) (Eq. 19)

ρ = density of suspension = density of water (kg/m³)

v_R = velocity of paddle relative to fluid (m/s) (Eq. 20)

$$C_{DP} = \frac{24}{Re} + \frac{4}{\sqrt{Re}} + 0.4 \tag{17}$$

Where, Re = Reynolds number (Eq. 18)

$$Re = \frac{D^2 N \rho}{\mu} \tag{18}$$

Where, D = diameter of impeller (0.009 m)

N = impeller's rotational speed (revolutions per second)

$$A_p = L \times W \tag{19}$$

Where, L = length of paddle

W = width of paddle

$$v_R = 2\pi(L/2)N \times 0.75 \tag{20}$$

0.75 = relative velocity of paddle with respect to fluid
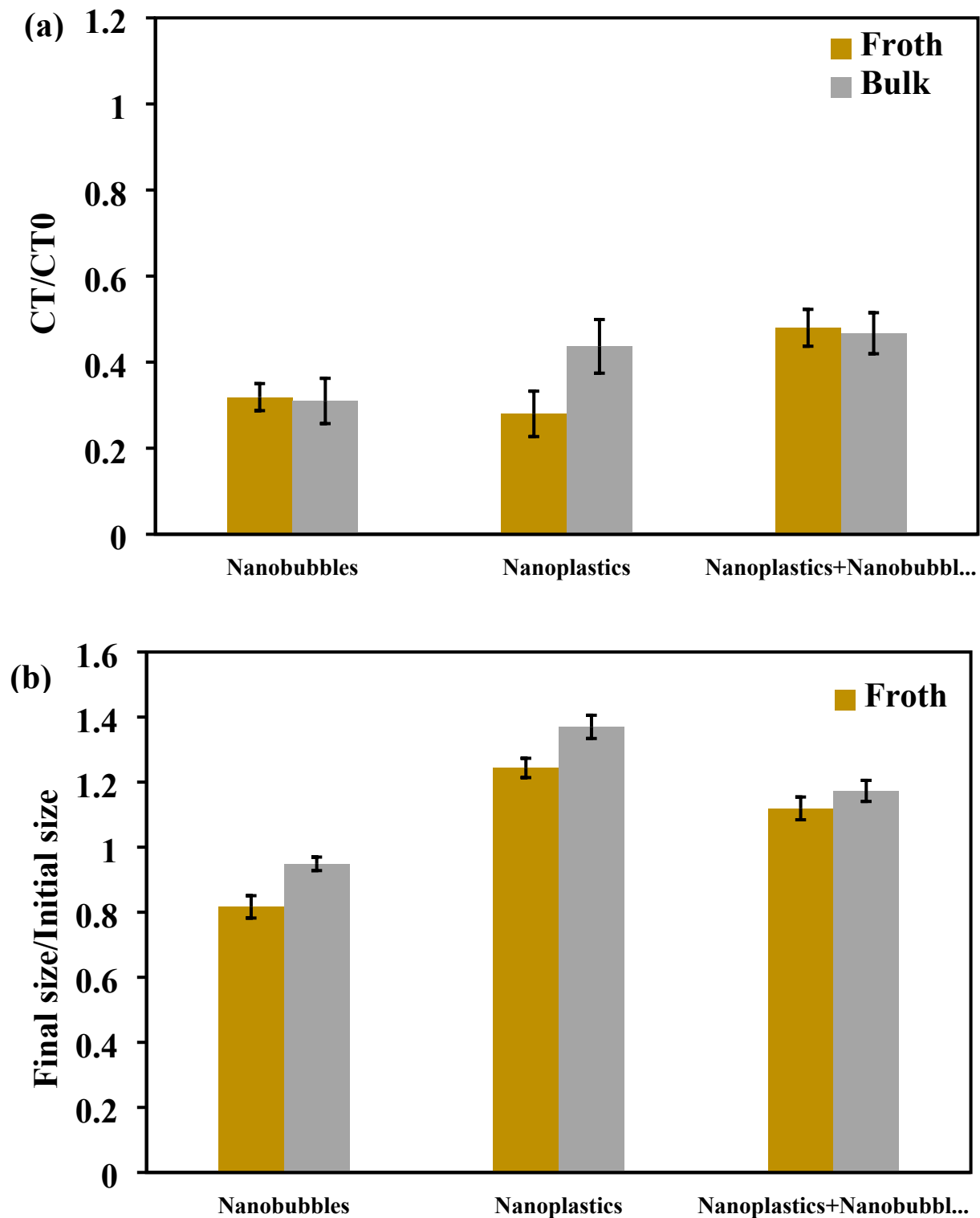
Fig. S5. (a) Normalized number concentration from NTA, and (b) Normalized average particle size distributed at the meniscus and bulk phase of DI water and nanobubble solution under static conditions (pH = 3, stirring speed = 0 RPM, 5 min).

Text S5. Findings from the effect of stirring velocity

The velocity gradients at 400 and 100 rpm are 271 and 39 s^{-1} respectively. Thus, the collision frequency between NPs and nanobubbles at 400 and 100 rpm was 2.8×10^{-11} cm^3 s^{-1} and 2.0×10^{-11} cm^3 s^{-1} respectively. The rate of attachment was computed as 4.2×10^4 cm^{-3} s^{-1} and 3.0×10^4 cm^{-3} s^{-1} respectively, assuming 100% attachment. A 69% of the NPs were removed at 100 rpm compared to 60% at 400 rpm based on mass concentration in the subnatant. The floc size in the float and subnatant at 100 rpm was also 28% and 13% larger respectively than that of 400 rpm. This slight increase in float mass concentration (Fig. S6a) and number concentration (Fig. S6b) at 100 rpm stirring suggests that the increased agitation and vortex formation may be re-introducing some NPs into the water column at 400 rpm. Moreover, the larger floc size at 100 rpm compared to 400 rpm implies better NP-nanobubble attachment at 100 rpm.

(b)

(c)
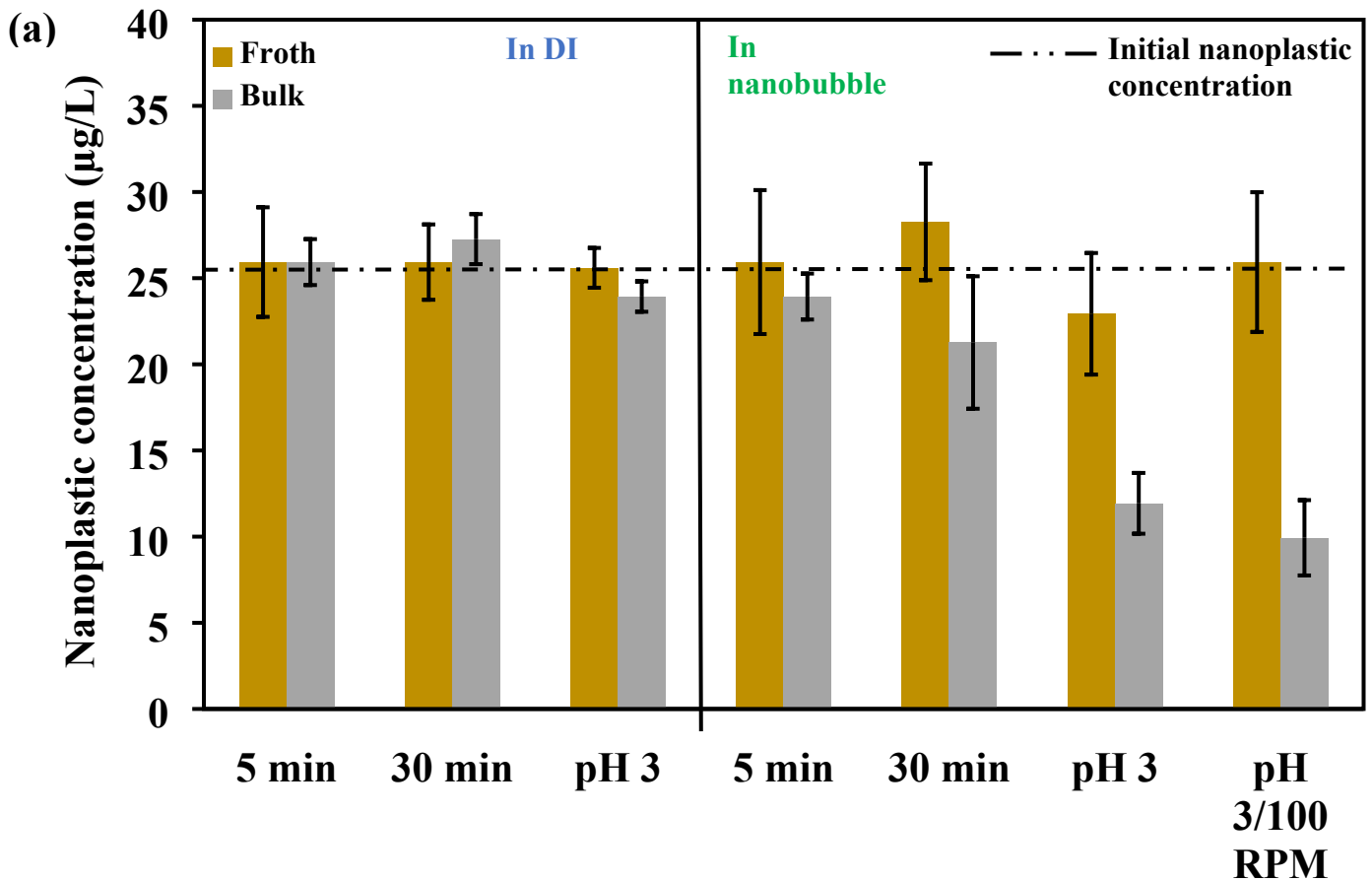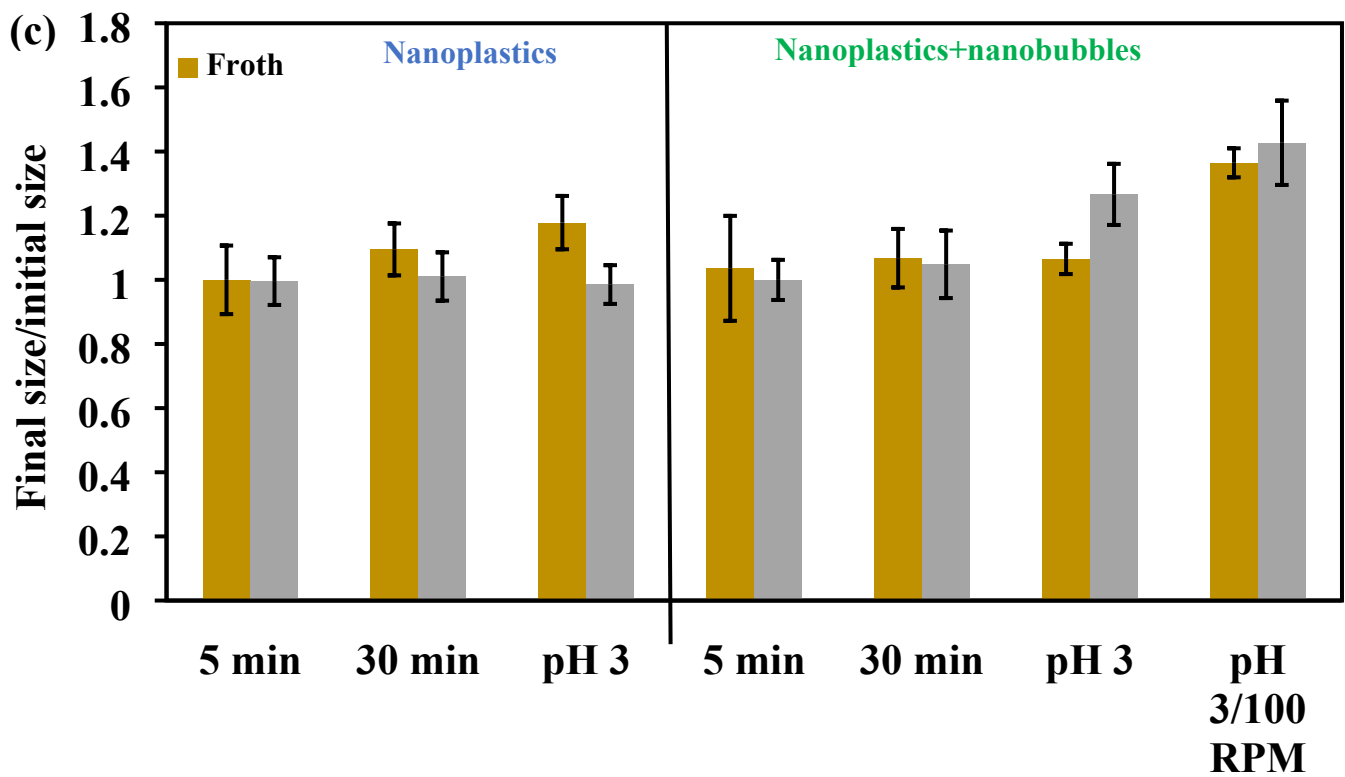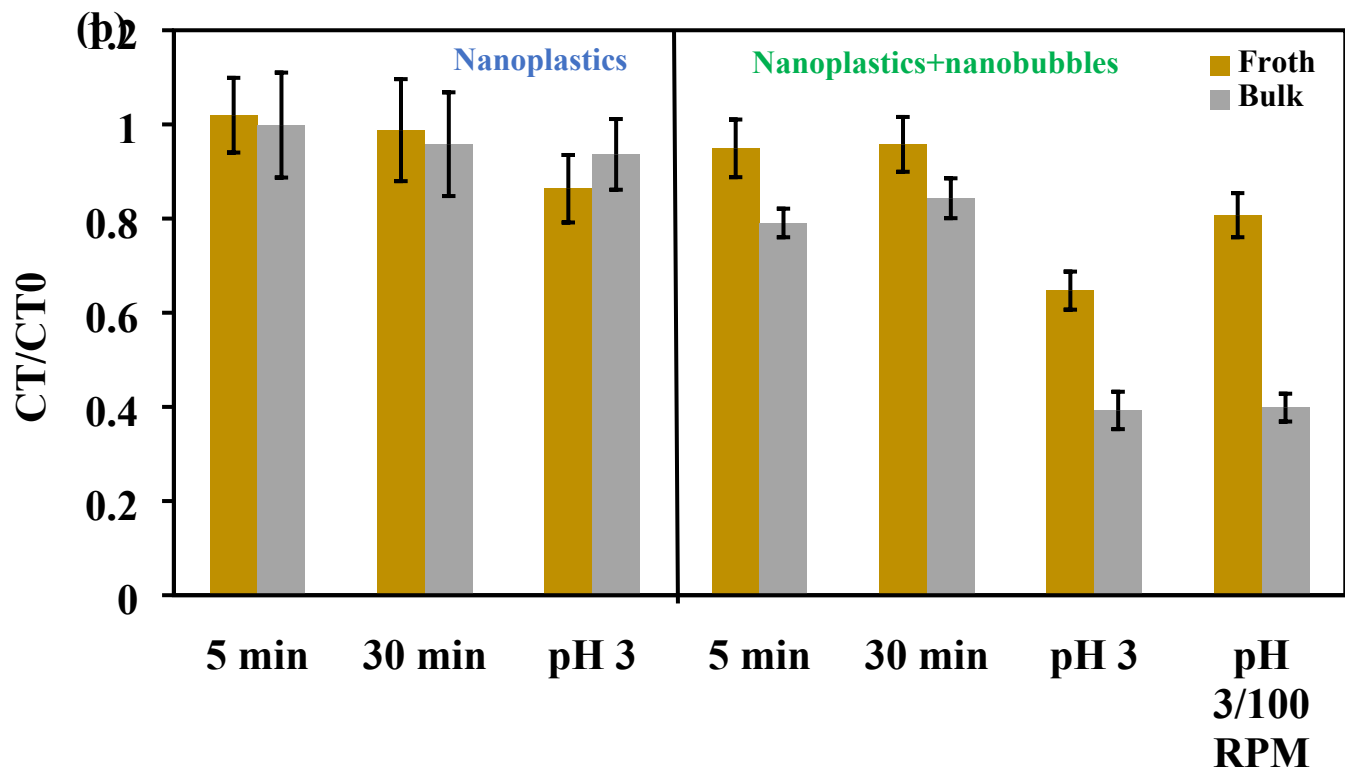
Fig. S6. (a) Mass concentration from UV-vis spectroscopy, (b) normalized number concentration from NTA, and (c) normalized average size of nanoplastics from NTA distributed at the meniscus and bulk phase of DI water and nanobubble solution after stirring (stirring time = 5 min unless stated otherwise, stirring speed = 400 rpm unless stated otherwise, pH is unmodified unless stated otherwise).

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