# Unbound Nanoparticle Release from Tire Wear

Greg Beaucage
Prof. Chemical and Materials Engineering
University of Cincinnati

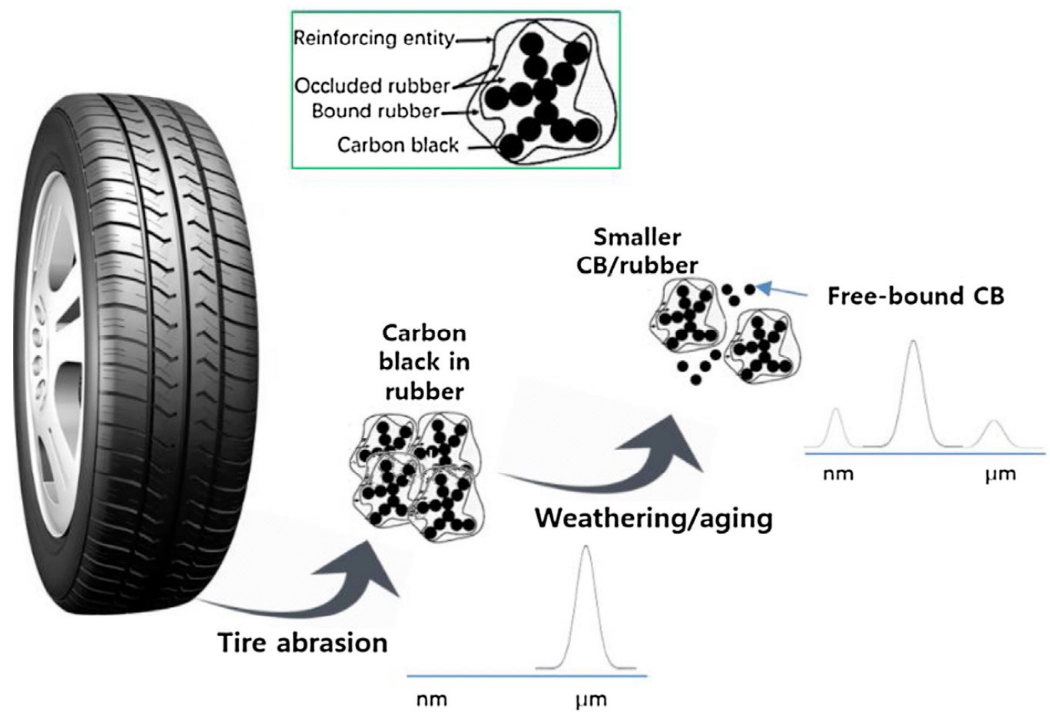
Ed Terrill
Senior Scientist
Akron Rubber Development Laboratory

Mingming Lu
Prof. Environmental Engineering, UC

Dr. Kabir Rishi
Scientist NIOSH/UC

Dr. Pramod Kulkarni
Senior Scientist NIOSH/HELD/CDC

gbeaucage@gmail.com
513 373-3454




Kim J, Yang SI, Moon H, Hong J, Hong J, Choi W, Son H, Lee B-C, Kim G-B, Kim Y *Potential release of nano-carbon black from tire-waer particles through the weathering effect* J. Ind. Eng. Chem. **96** 322-329 (2021).

Greg Beaucage Univ. Cincinnati beaucag@uc.edu

Are nano-tire wear particles (nTWPs)
a problem?

- Overview of the topic
- Recent literature investigations
- Our recent measurements

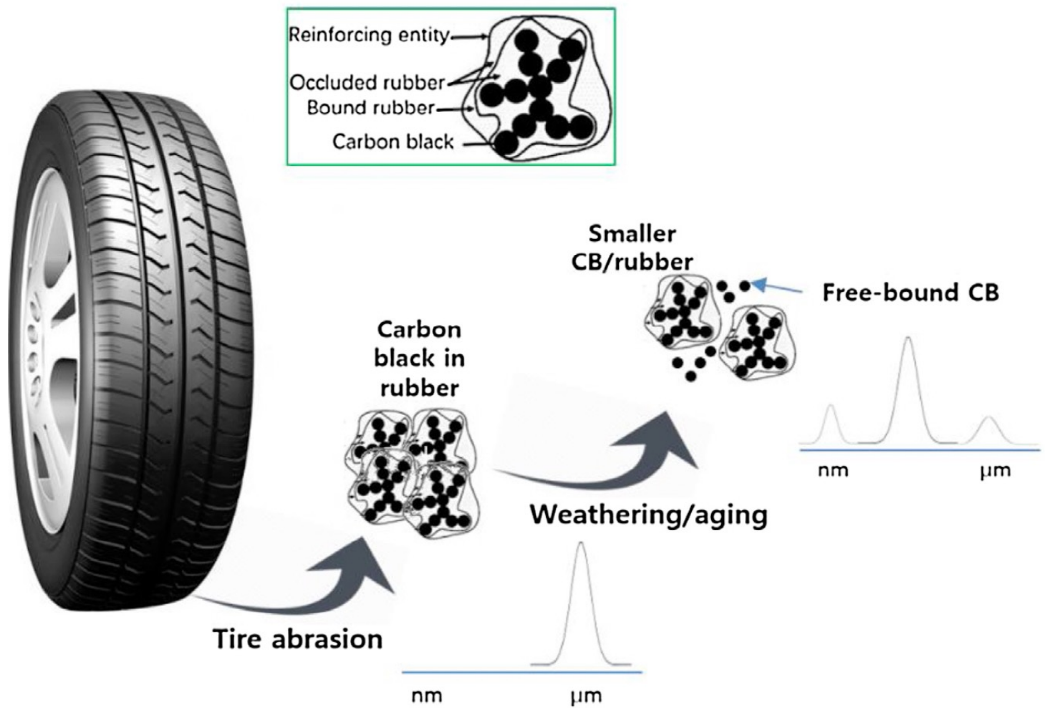



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**Fig. 1** Composition of reference passenger car tire in the European Union. Data (in wt%) from OECD [5]

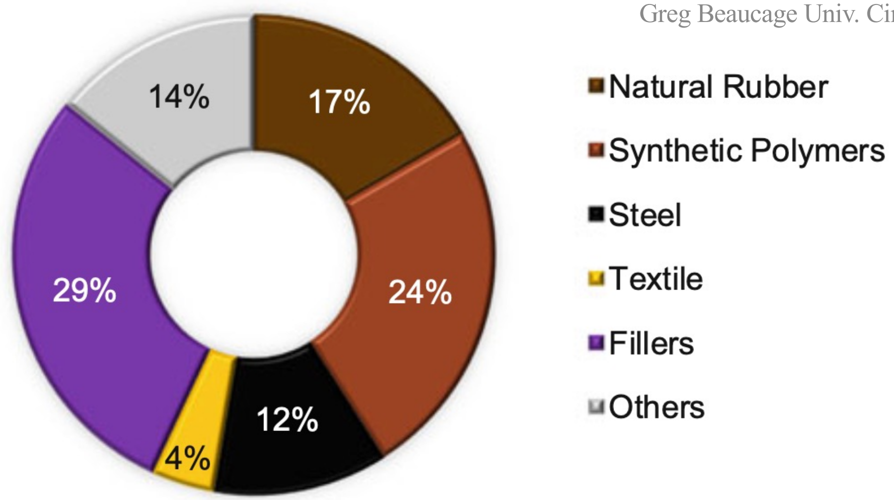
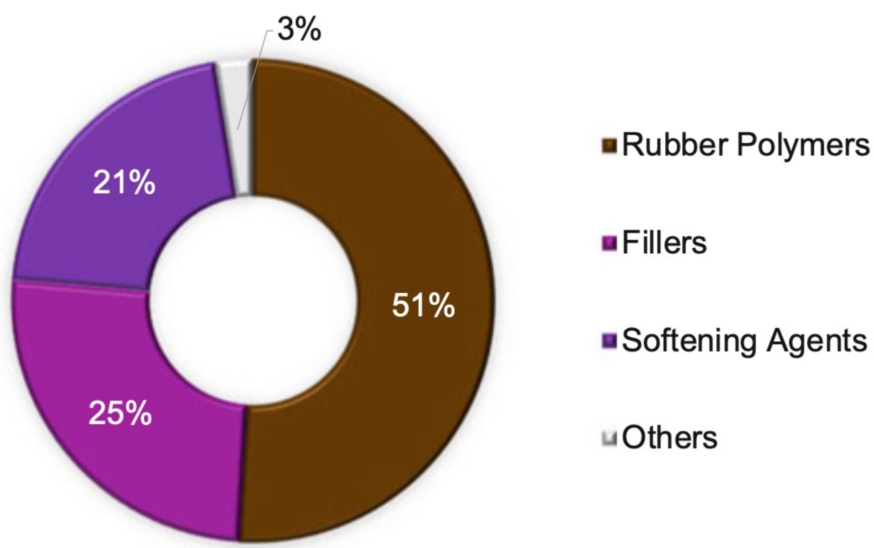



**Fig. 2** Example of a tire-tread composition. Data (in wt%) from Winther and Slentø [9]




Gieré R, Dietze V *Tire-Abrasion Particles in the Environment* Adv. Polym. Sci. **118** 12 (2022).

**Table 6.** Calculation of the amount of tyre wear and tear in Germany [37,38].

| | Wear (mg/km) [37] | Total Wear 2001/2002 tonnes/year [37] | Total Mileage in 2013 ($\times 10^6$ km) [38] | Total Wear 2013 tonnes/year |
|---|---|---|---|---|
| Moped | 22.5 | 88 | 4700 | 106 |
| Motorcycle | 45 | 621 | 12,300 | 689 |
| Passenger car | 90 | 46,017 | 615,100 | 55,359 |
| Bus | 700 | 2590 | 3300 | 2310 |
| Lorry | 700 | 43,540 | 64,300 | 45,010 |
| Articulated lorry | 1200 | 16,440 | 16,700 | 20,040 |
| Other | 180 | 2124 | 9300 | 1674 |
| Total | | 111,420 | 725,700 | 125,188 |

Kole PJ, Löhr AJ, Van Belleghem FGAJ, Ragas AMJ *Wear and Tear of Tyres: A Stealthy Source of Microplastics in the Environment* Int. J. Env. Res. Pub. Health **14** 1265 (2017).

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Times 7 for US?

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**Table 16.** Weight of Internal Combustion Engine (ICE)-cars compared to the electric version. The weight of the petrol car includes a half full tank.

| Petrol Version | Weight [kg] | E-Version | Weight [kg] | Extra Weight E-Version |
|---|---|---|---|---|
| Volkswagen high Up! petrol | 958 | Volkswagen e-Up! | 1114 | 16% |
| Volkswagen Golf 1.4 TSI | 1205 | Volkswagen E-Golf | 1485 | 23% |
| Ford Focus | 1380 | Ford Focus Electric | 1674 | 21% |
| Mercedes-Benz B 250 | 1465 | Mercedes-Benz B 250 e | 1725 | 18% |

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**Weight is distributed lower in the vehicle, cornering is sharper**

**Torque is significantly greater especially from a dead start**

**Chevy Bolt (micro commuter car) 0-60 in < 4 seconds**

**EV tires may contain more silica**

Kole PJ, Löhr AJ, Van Belleghem FGAJ, Ragas AMJ *Wear and Tear of Tyres: A Stealthy Source of Microplastics in the Environment* Int. J. Env. Res. Pub. Health **14** 1265 (2017).

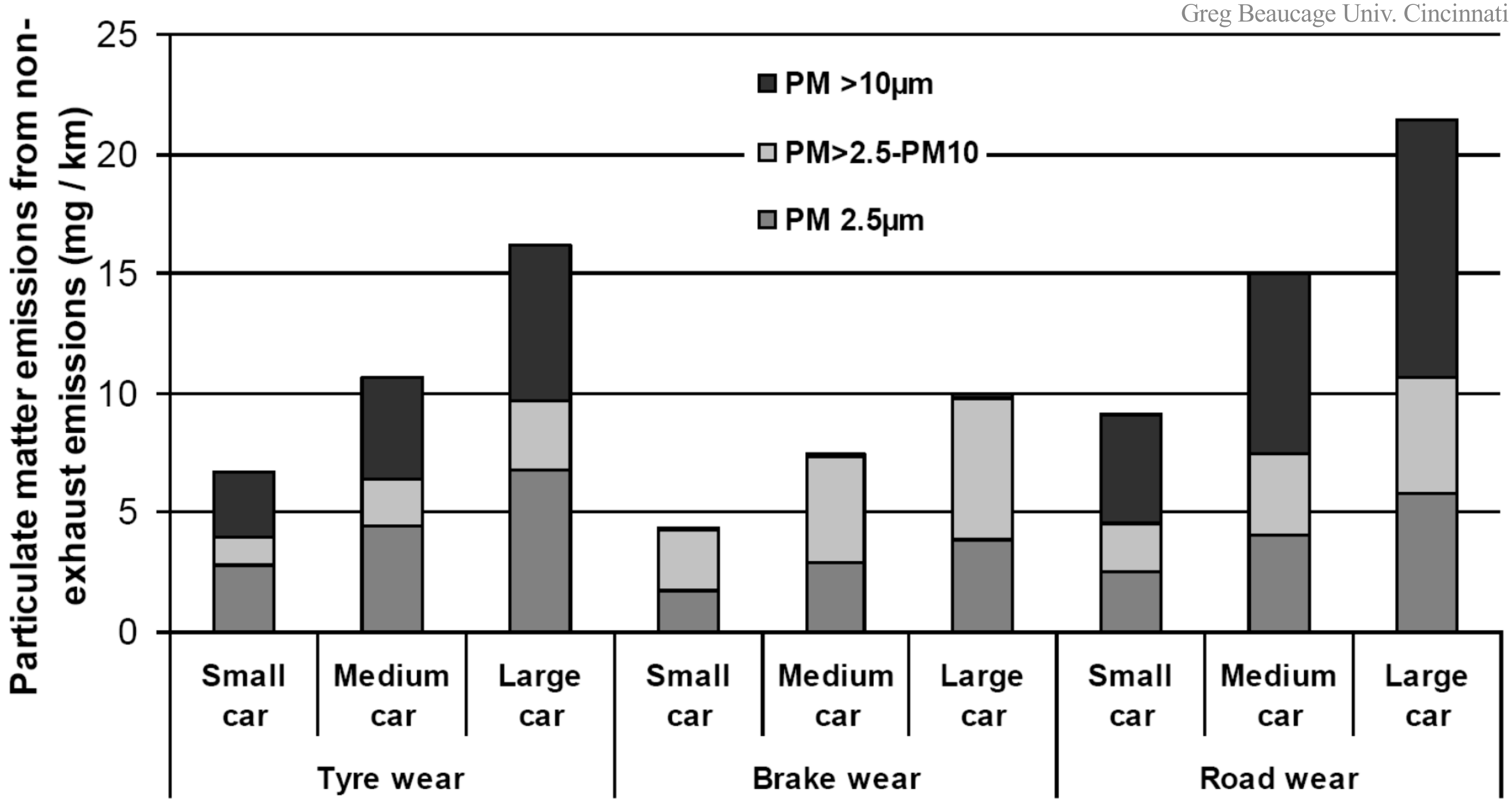


**Figure 4.** Non-exhaust particulate matter (PM) emissions by source and car size, from Simons [113] based on Ntziachristos and Boulter [117].

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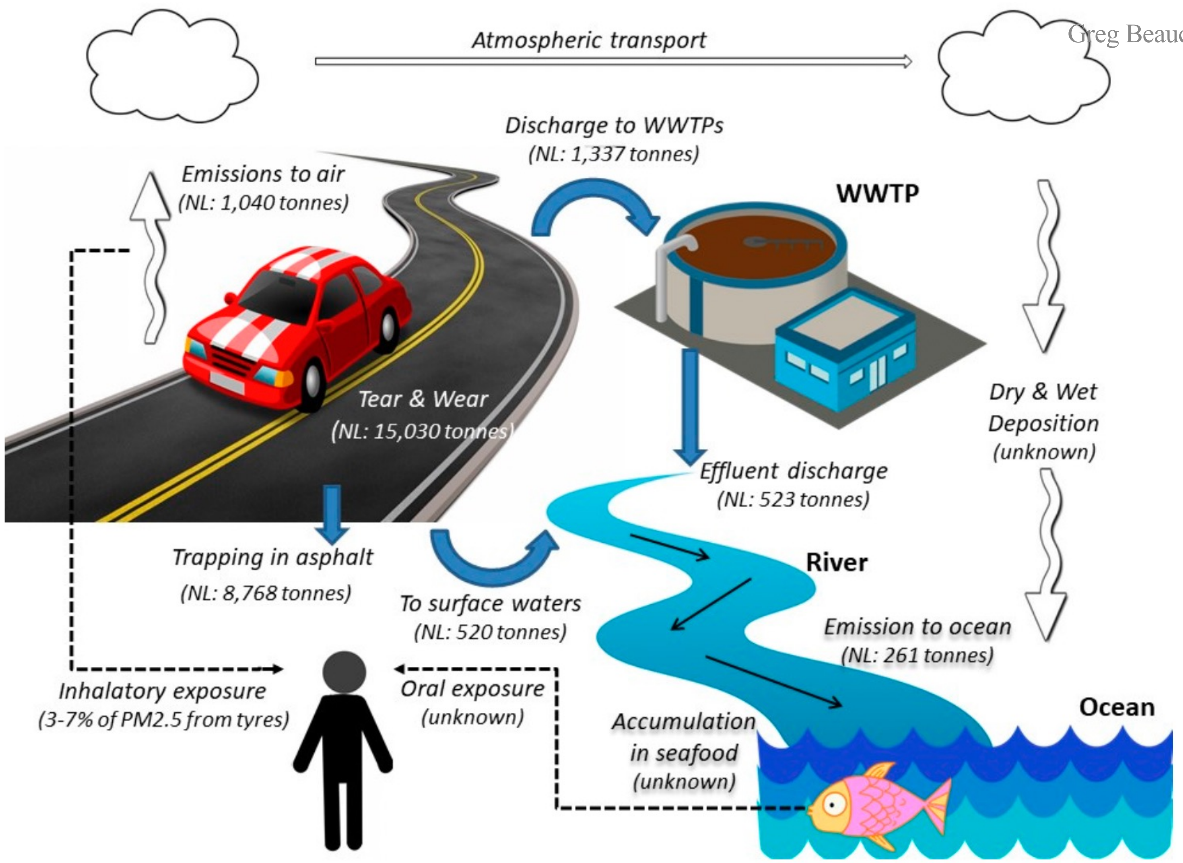


**Figure 3.** Distribution of the tyre wear and tear over the compartments. WWTP: waste water treatment plants; NL: The Netherlands.

Kole PJ, Löhr AJ, Van Belleghem FGAJ, Ragas AMJ *Wear and Tear of Tyres: A Stealthy Source of Microplastics in the Environment* Int. J. Env. Res. Pub. Health **14** 1265 (2017).

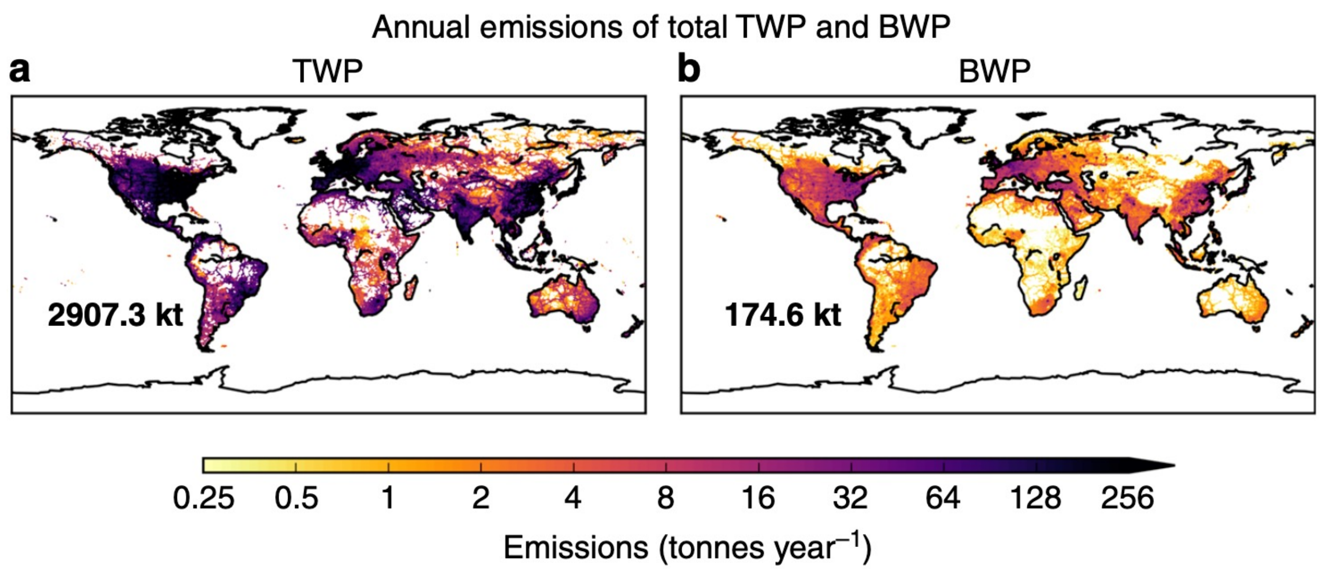


**Fig. 1 Annual gridded emissions of road microplastics.** Global annual emissions of total road microplastics (tyre wear particles, TWPs, in **a**, and brake wear particles, BWPs, in **b**). TWP emissions are the average of the calculated emissions using the $CO_2$ ratio method and the GAINS model. Bold numbers at the left bottom of each panel represent the annual emissions of total TWPs and BWPs from road vehicles for 2014, which were estimated to be 2907 and 174.6 kt, respectively.

Evangeliou N, Grythe H, Klimont Z, Heyes C, Eckhardt S, Lopez-Aparicio S, Stohl A *Atmospheric transport is a major pathway of microplastics to remote regions* Nature **11** 3381 (2020).

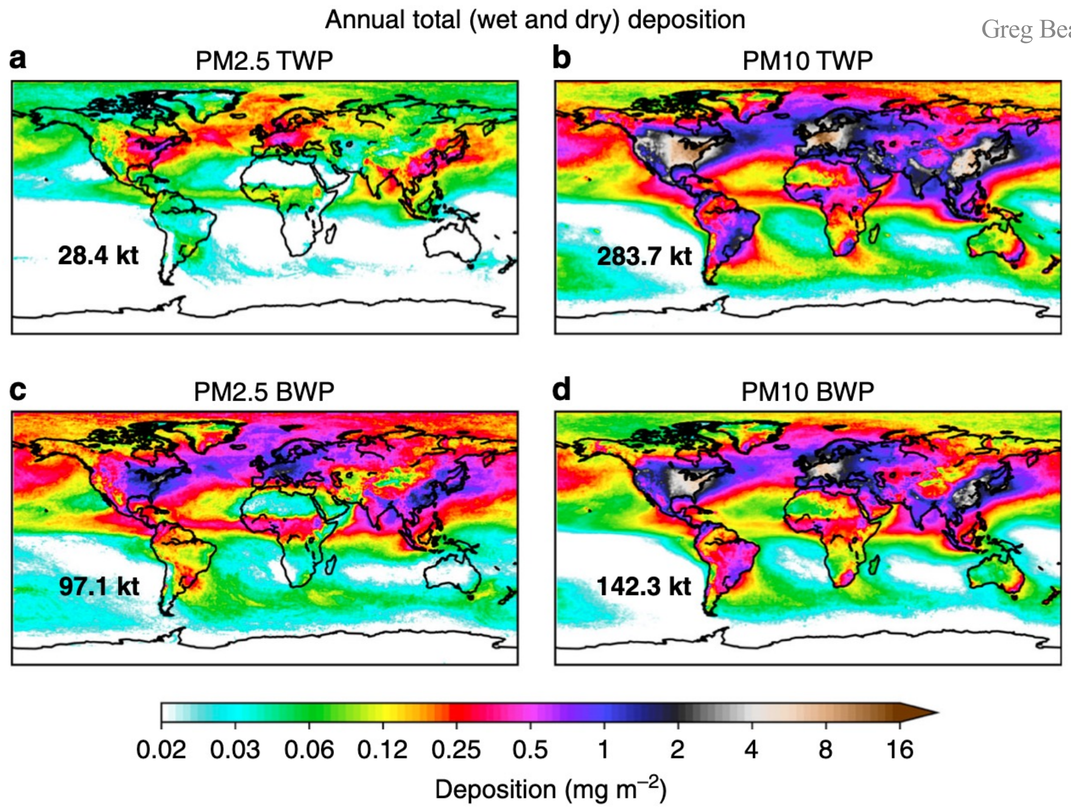


**Fig. 2 Wet and dry deposition of road microplastics.** Annual total (wet and dry) deposition of tyre wear particles (TWPs) and brake wear particles (BWPs) in PM2.5 and PM10 size classes, respectively. The projected deposition has been calculated as the geometric mean of all simulations using TWP emissions estimated using the $CO_2$ ratio method and the GAINS model and using BWP emissions calculated from the GAINS model, respectively. The simulations comprise 120 ensemble members with different assumption for the airborne fraction (five for each of the PM2.5 and PM10 fractions), particle size distribution (eight for each of the PM2.5 and PM10 fractions) and CCN/IN (cloud condensation nuclei/ice nuclei) efficiency (three different sets of scavenging coefficients per fraction) following a log-normal distribution (see "Methods"). Bold numbers at the left bottom of each panel represent the annual total deposition of TWPs and BWPs from road vehicles in PM2.5 and PM10 sizes for year 2014.

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**Laboratory studies of TWPs**

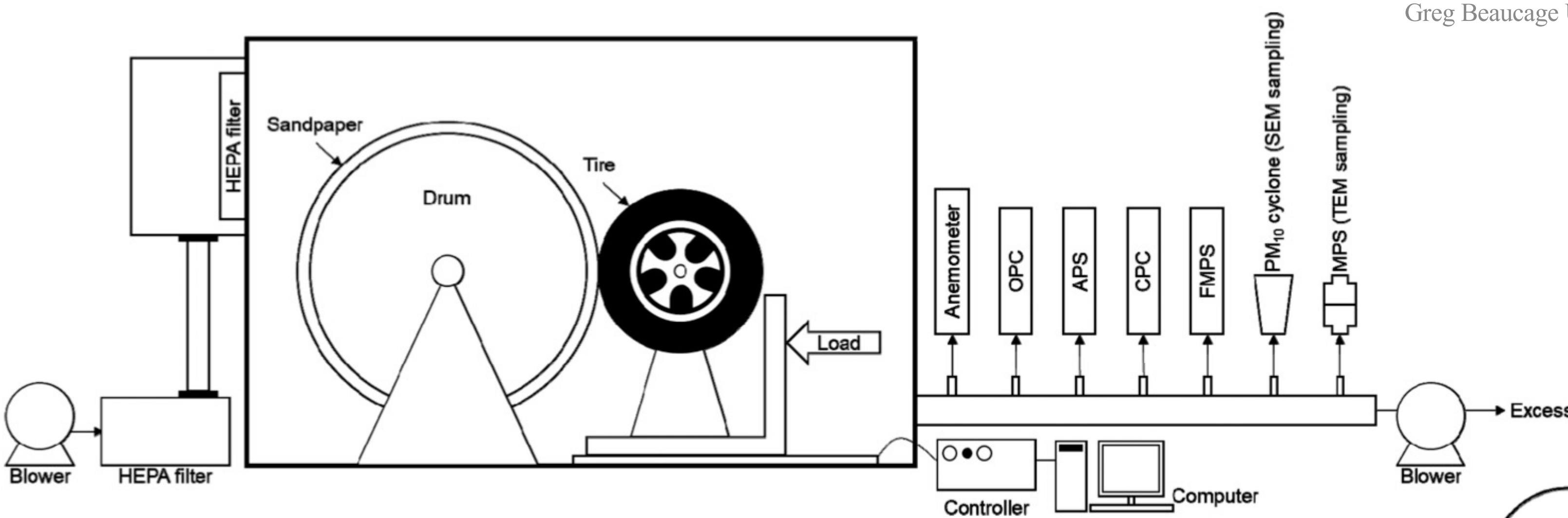
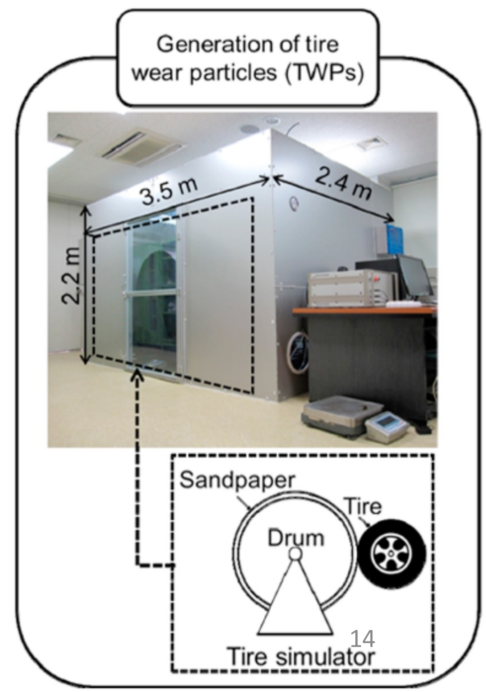
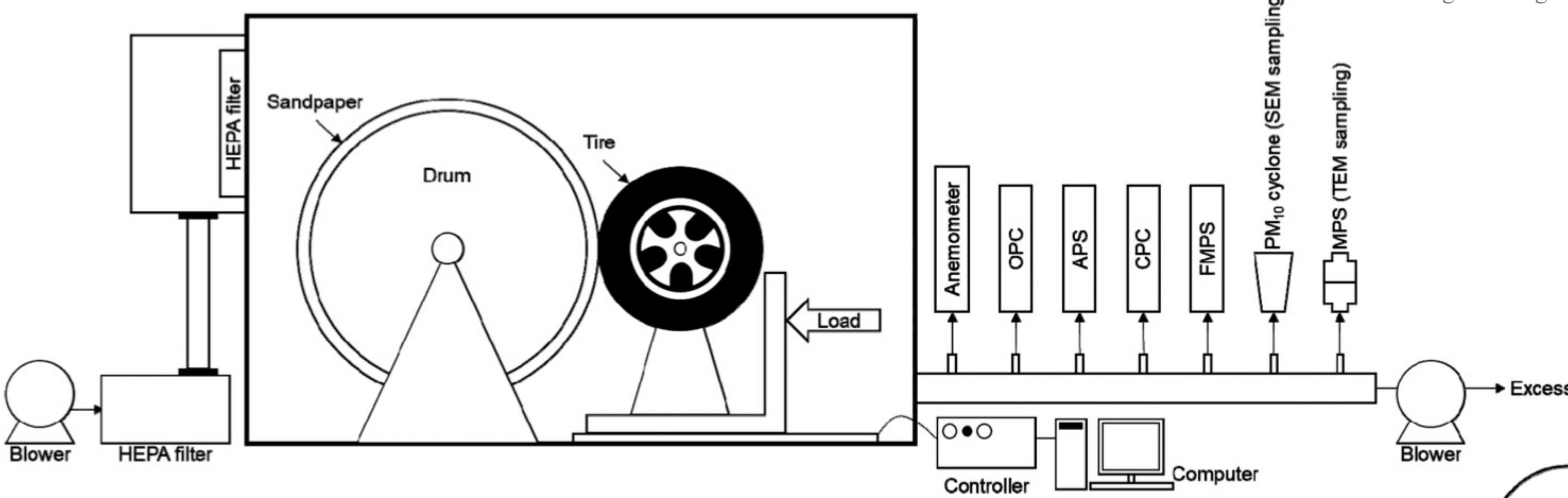



**Figure 1.** Schematic of the tire simulator operated within the enclosing chamber and the measurement setup.




OPC optical particle counter
APS aerodynamic particle sizer
CPC condensation particle counter
FMPS fast mobility particle sizer
MPS mini-particle sizer

Kim G, Lee, S *Characteristics of Tire Wear Particles Generated by a Tire Simulator under Various Driving Conditions* Environ. Sci. Technol. **52** 12153-12161 (2018).

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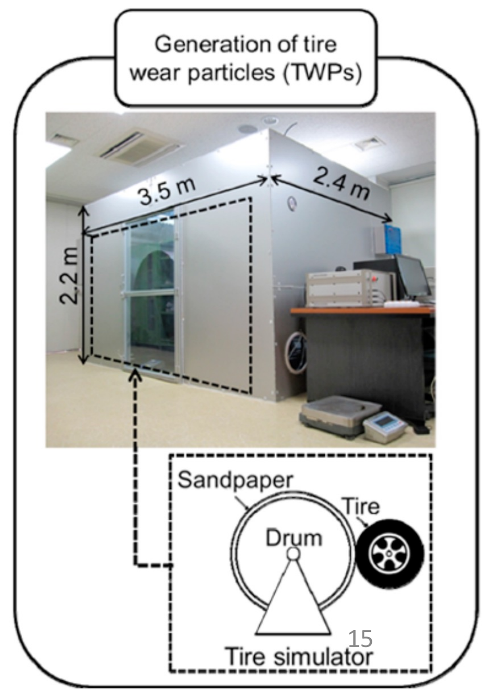


# Most particle emissions occur on cornering and breaking

# This type device does not these conditions

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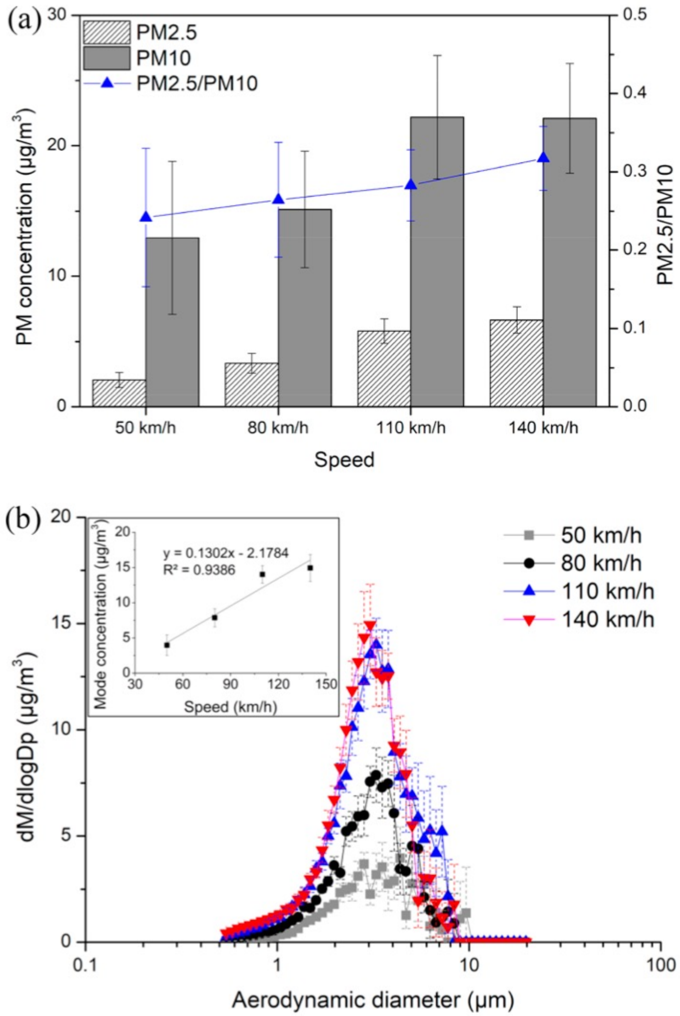



**Figure 2.** (a) Average particulate $(PM)_{2.5}$ and $PM_{10}$ concentrations, $PM_{2.5}/PM_{10}$ ratio, and (b) mass size distributions by mode concentration under constant driving speeds (50, 80, 110, 140 km $h^{-1}$).

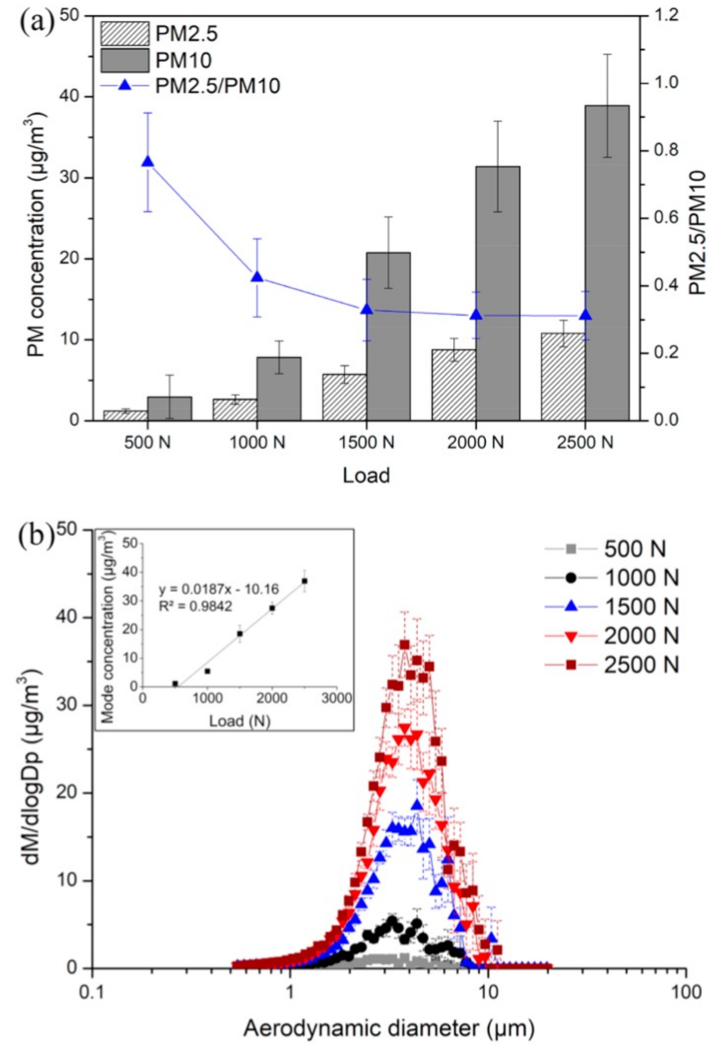
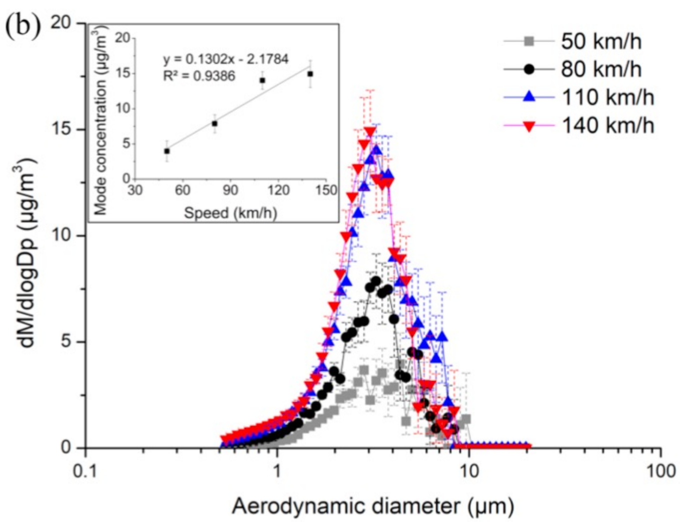



**Figure 3.** (a) Average $PM_{2.5}$ and $PM_{10}$ concentrations, $PM_{2.5}/PM_{10}$ ratio, and (b) mass size distributions by mode concentration under consistent loads (500, 1000, 1500, 2000, and 2500 N).

# APS is limited to particles larger than ~1 µm
# Mass/volume emphasizes large particles

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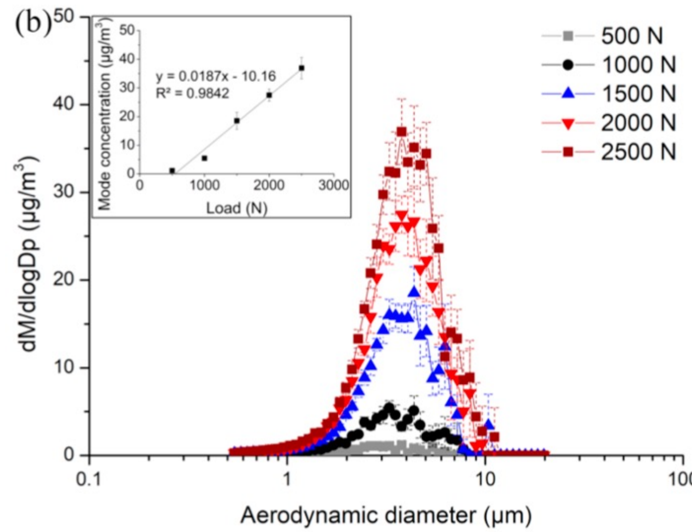


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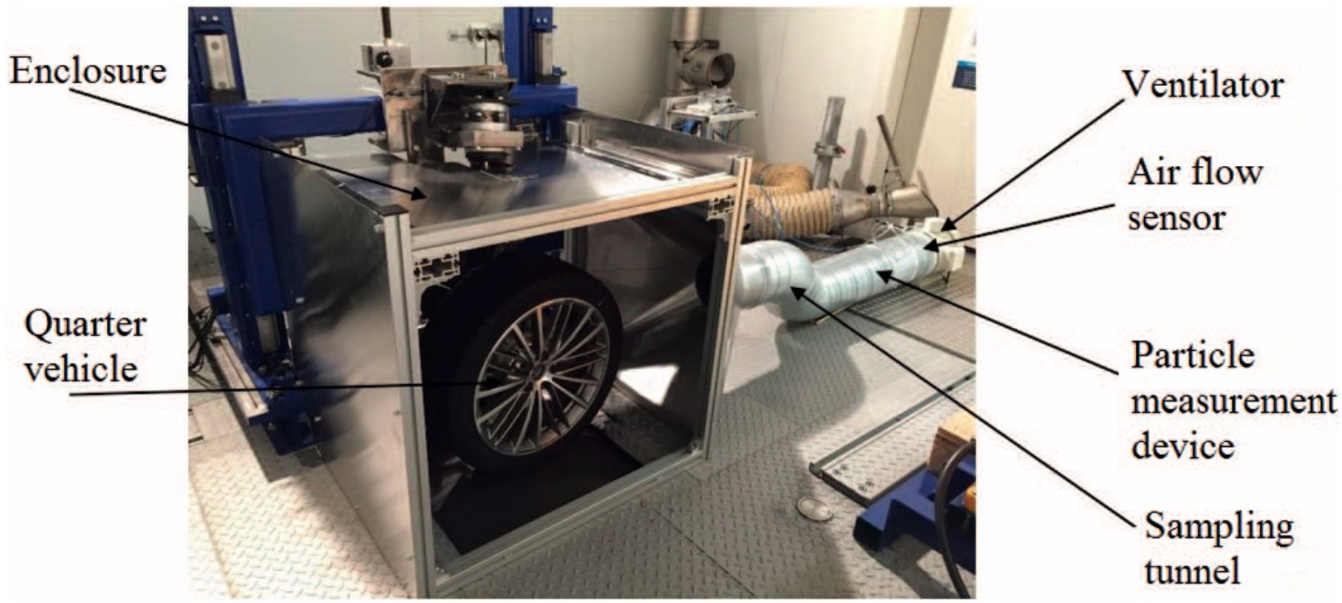
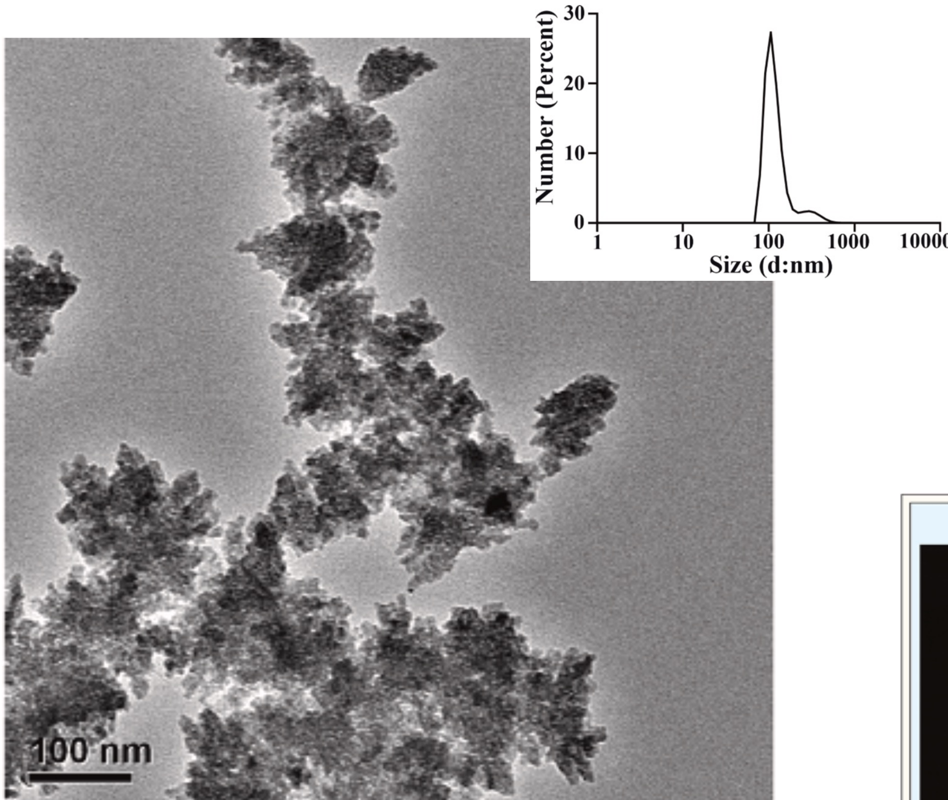
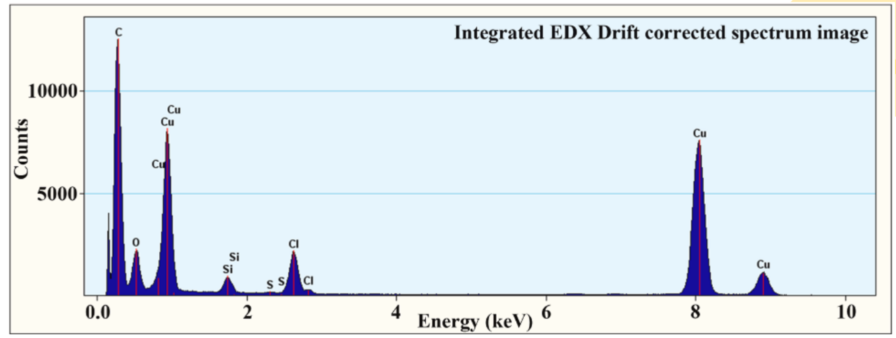


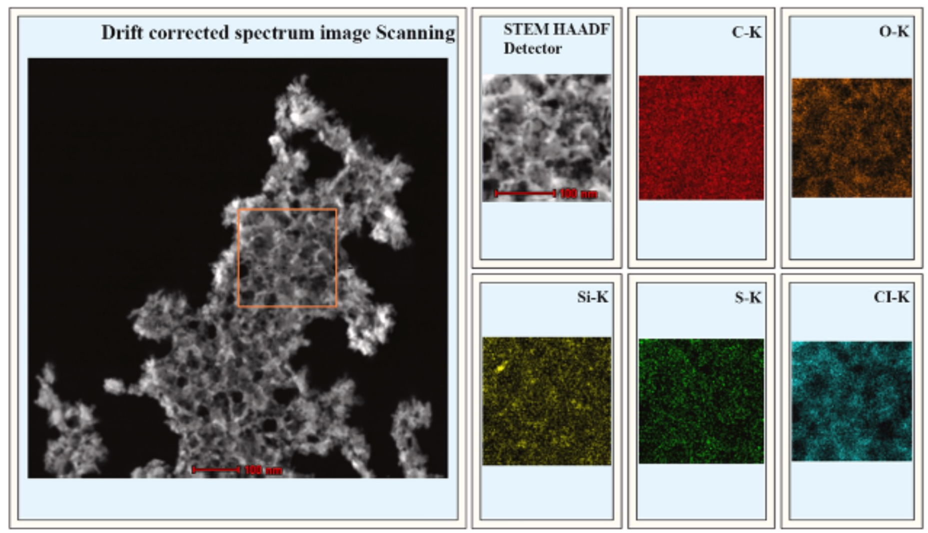

FIG. 1 — *New developed box built in the quarter car system over the chassis dynamometer.*

Dalmau ME, Augsburg K, Wenzel F, Ivanov V *Tire Particle Emissions: Demand on Reliable Characterization* Tire Sci. Tech. TSTCA **48** 107-122 (2020).

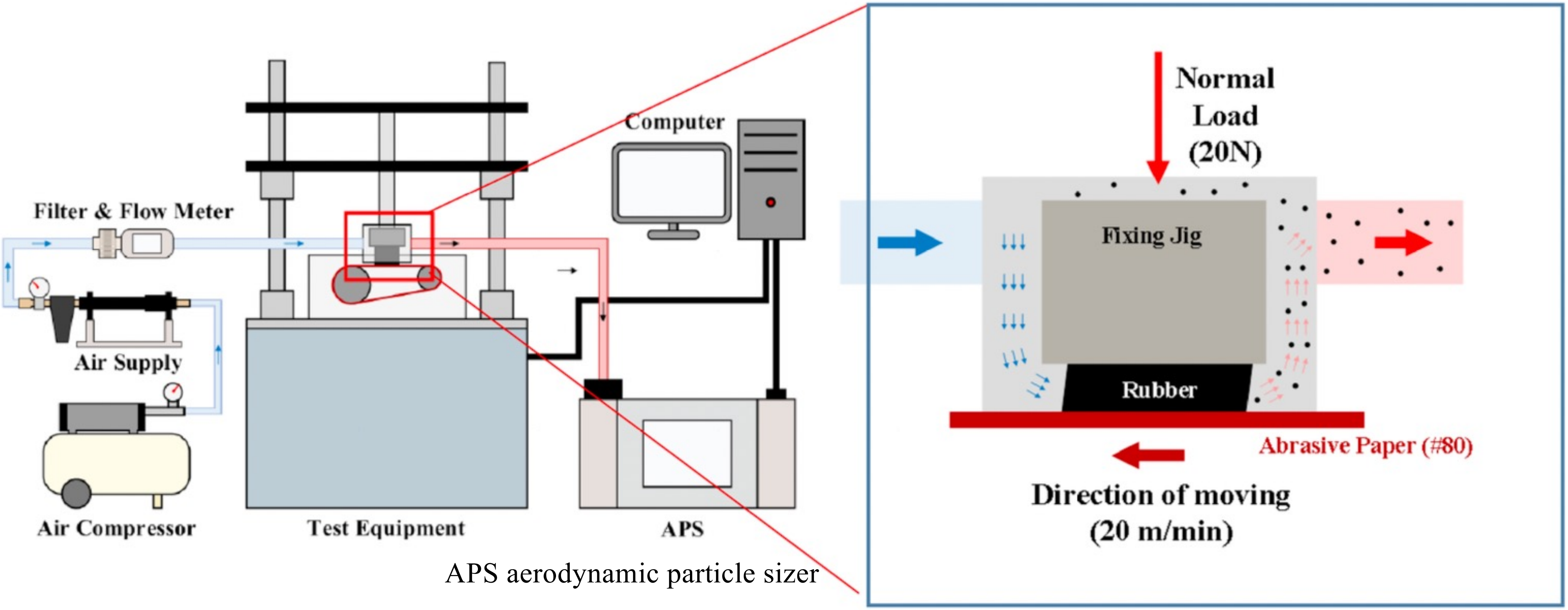
Karlsruhe




Li Y, Shi T, Li X, Sun H, Xia X, Ji X, Zhang J, Liu M, Lin Y, Zhang R, Zheng Y, Tang J *Inhaled tire-wear microplastic particles induced pulmonary fibrotic injury via epithelial cytoskeleton rearrangement* Env. Int. **164** 107257 (2022).

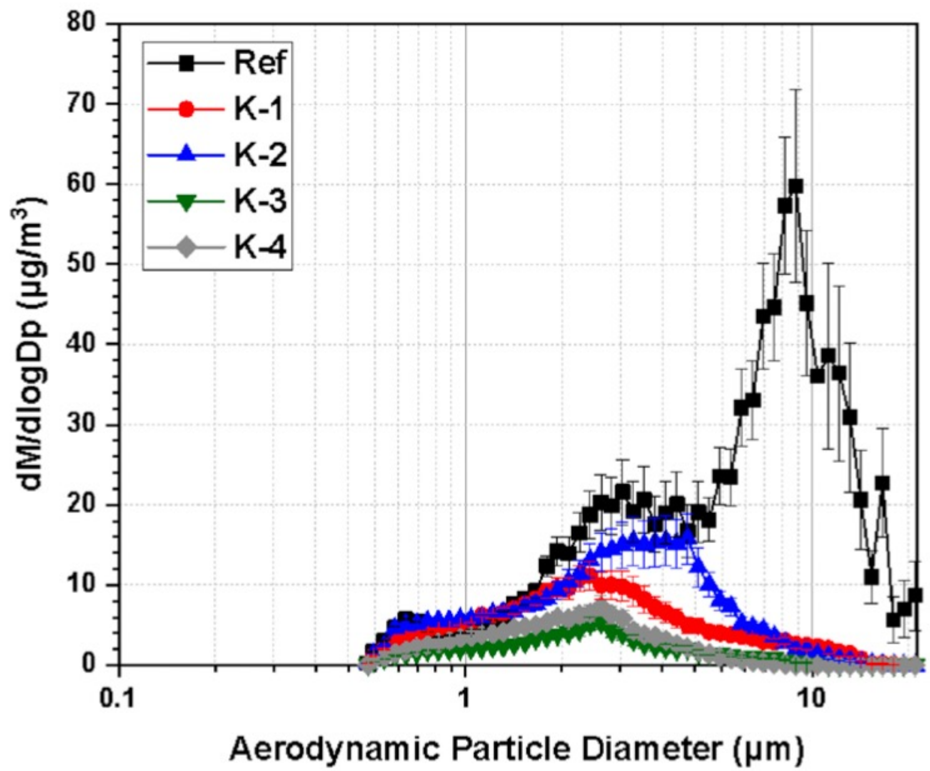



Ha, JU, Lee HH, Ryu G, Lee P-C, Ko YK, Jeoung SK *Trends in particulate matter generation from truck and bus radial tires using a wear tester* J. Appl. Polym. Sci. **139** e52190 (2022)

**TABLE 2** Compound compositions (phr) and properties

| Material | K-0 (ref) | K-1 | K-2 | K-3 | K-4 | K-5 |
|---|---|---|---|---|---|---|
| NR[a] | 100 | 0 | 0 | 0 | 70 | 70 |
| NdBR40[b] | | 100 | | 70 | | |
| NdBR60[c] | 0 | 0 | 100 | 30 | 30 | 30 |
| Carbon black (N234) | 55 | 55 | 55 | 55 | 55 | |
| Carbon black (OCI-A) | | | | | | 55 |
| Common formulations | TDAE oil[d] (5), ZnO (4), Stearic acid (3), 6PPD (2), TMQ (1), Sulfur (1.3), TBBS (1), PVI (0.3) | | | | | |
| Bound rubber content (%) | 34.5 | 14.4 | 14.6 | 15.6 | 35.6 | 36.6 |
| Crosslink density ($10^{-5}$ mol/g) | 8.14 | 8.10 | 9.01 | 9.18 | 8.71 | 9.58 |
| DIN abrasion loss (mg) | 91.3 | 8.3 | 5.8 | 7.7 | 43.8 | 34.5 |
| Tan δ at 0°C | 0.181 | 0.138 | 0.121 | 0.119 | 0.161 | 0.157 |
| Tan δ at 60°C | 0.122 | 0.116 | 0.096 | 0.094 | 0.114 | 0.109 |

[a]Standard Vietnamese Natural Rubber SVR-10, dirt content of 0.1 wt%.
[b]Butadiene rubber with Mooney viscosity of 40.
[c]Butadiene rubber with Mooney viscosity of 60.
[d]Treated Distillate Aromatic Extracted.

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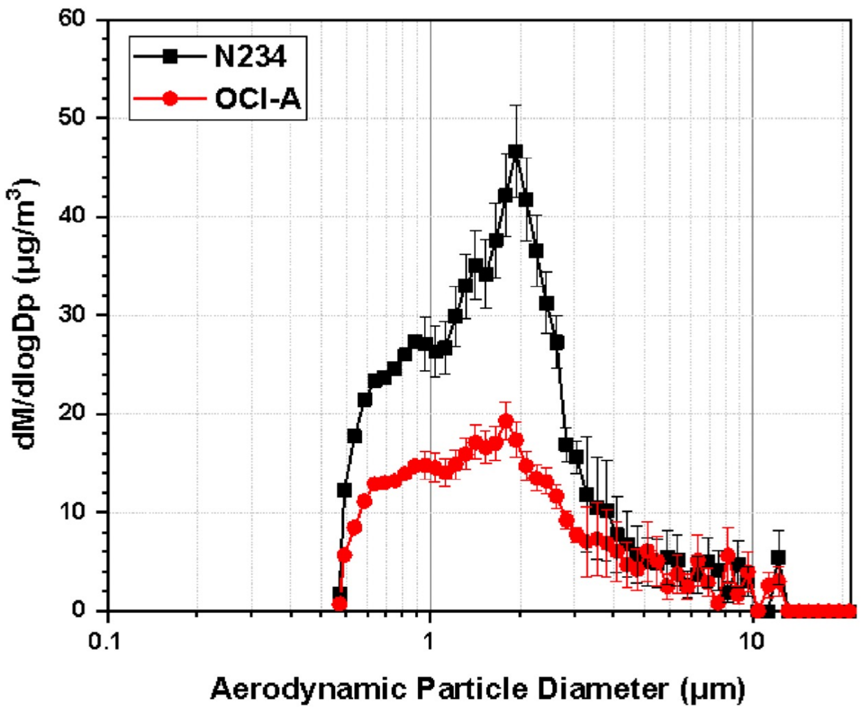


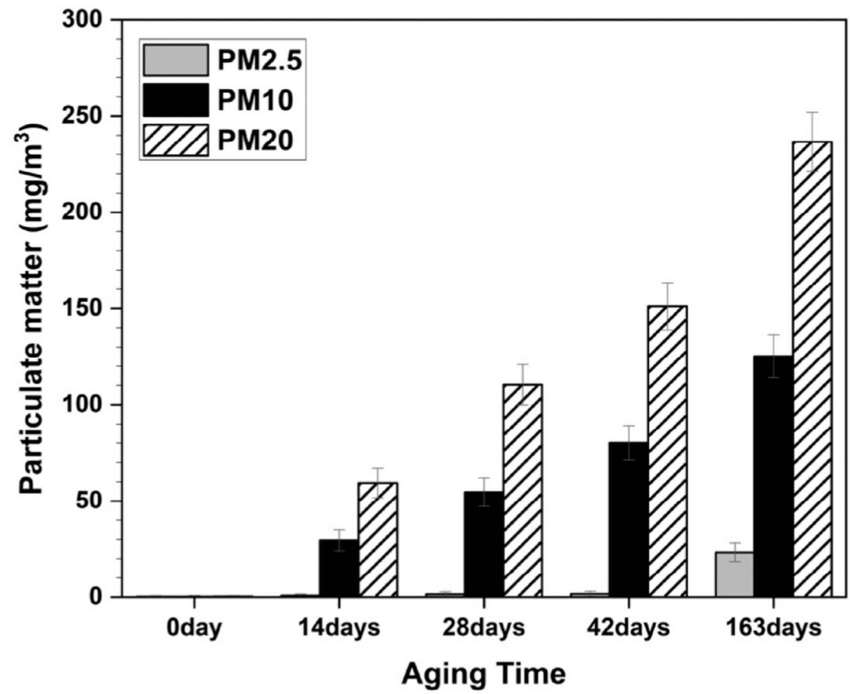


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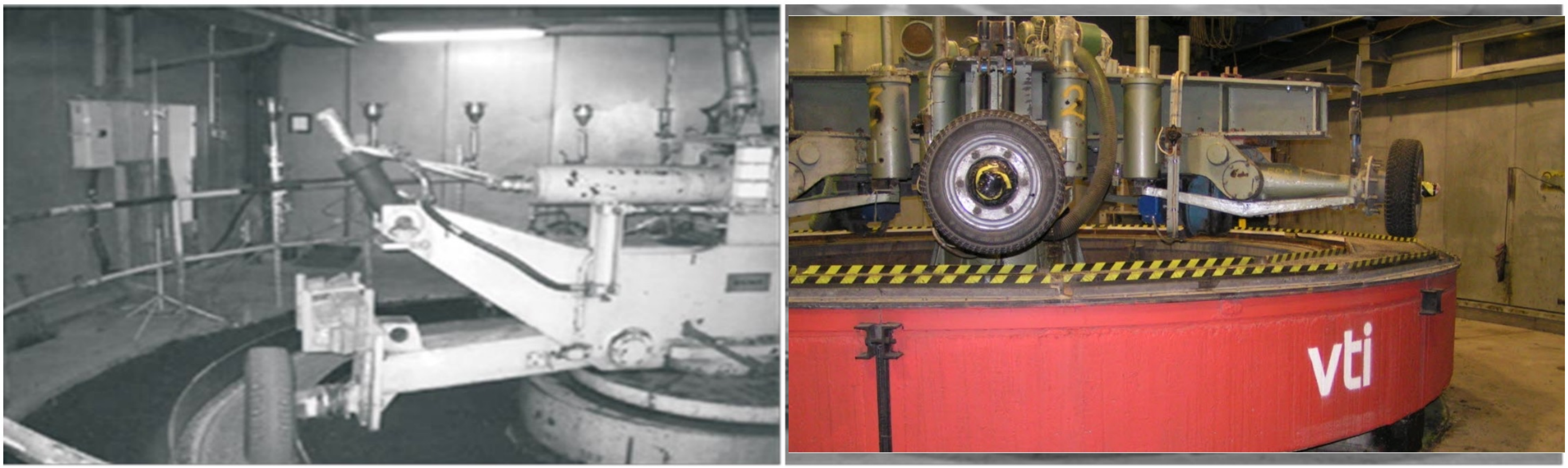
**Fig. 15: Test room with the rotating axle system in the foreground and the impactor inlets in the back (left) and the VTI road simulator (right). Sources: [Left - Kupiainen et al., 2005; Right - Gustafsson et al., 2008]**




Grigoratos T, Martini G *Non-exhaust traffic related emissions. Brake and tyre wear PM.* European Commission Joint Research Centre, Institute of Energy and Transport Science and Policy Reports: Publications Office of the European Union, Luxemborg (2014).

VTI = Swedish National Road and Transport Research Institute (VTI), SE-582 95 Linköping, Sweden

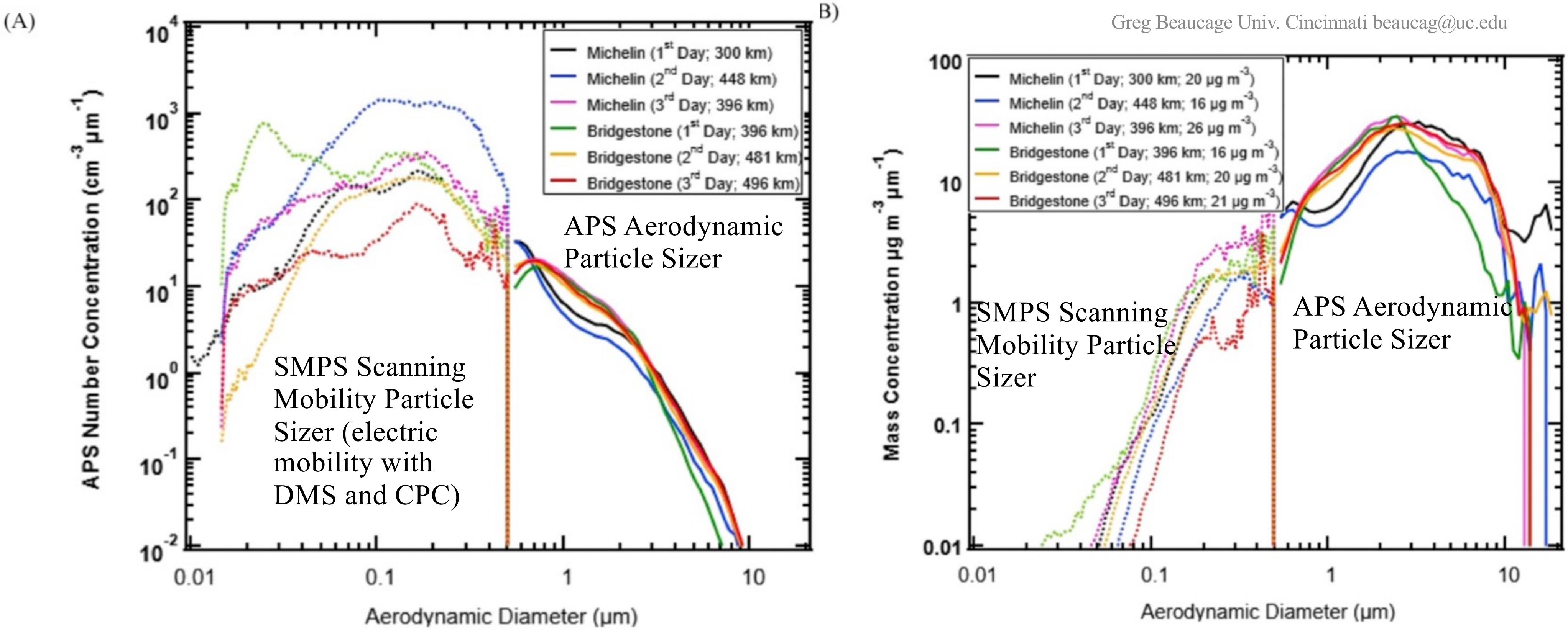
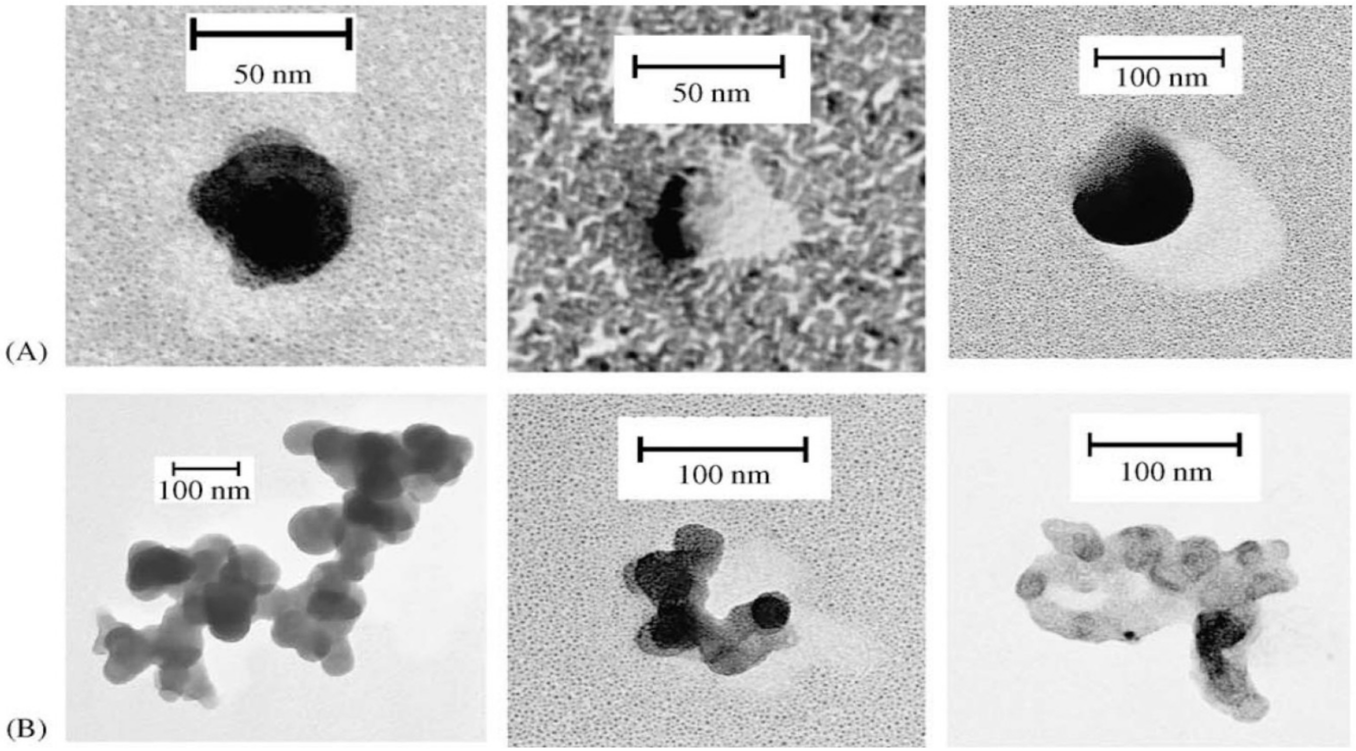



**Fig. 2.** Number (A) and mass (B) size distributions measured with the SMPS (dashed curves) and APS (solid curves) highlighting differences related to distance travelled by the same type of tyres (Michelin and Bridgestone).

Alves CA, Vicente AMP, Calvo AI, Baumgardner D, Amato F, Querol X, Pio C, Gustafsson M *Physical and chemical properties of non-exhaust particles generated from wear between pavements and tyres* **224** 117252 (2020). Used VTI simulator.

**Fig. 11: Examples of particles generated by the road simulator and collected after a DMA set at 40 nm. The images were taken with TEM. Source: [Dahl et al., 2006]**



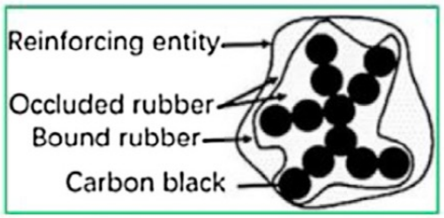


Grigoratos T, Martini G *Non-exhaust traffic related emissions. Brake and tyre wear PM.* European Commission Joint Research Centre, Institute of Energy and Transport Science and Policy Reports: Publications Office of the European Union, Luxemborg (2014).

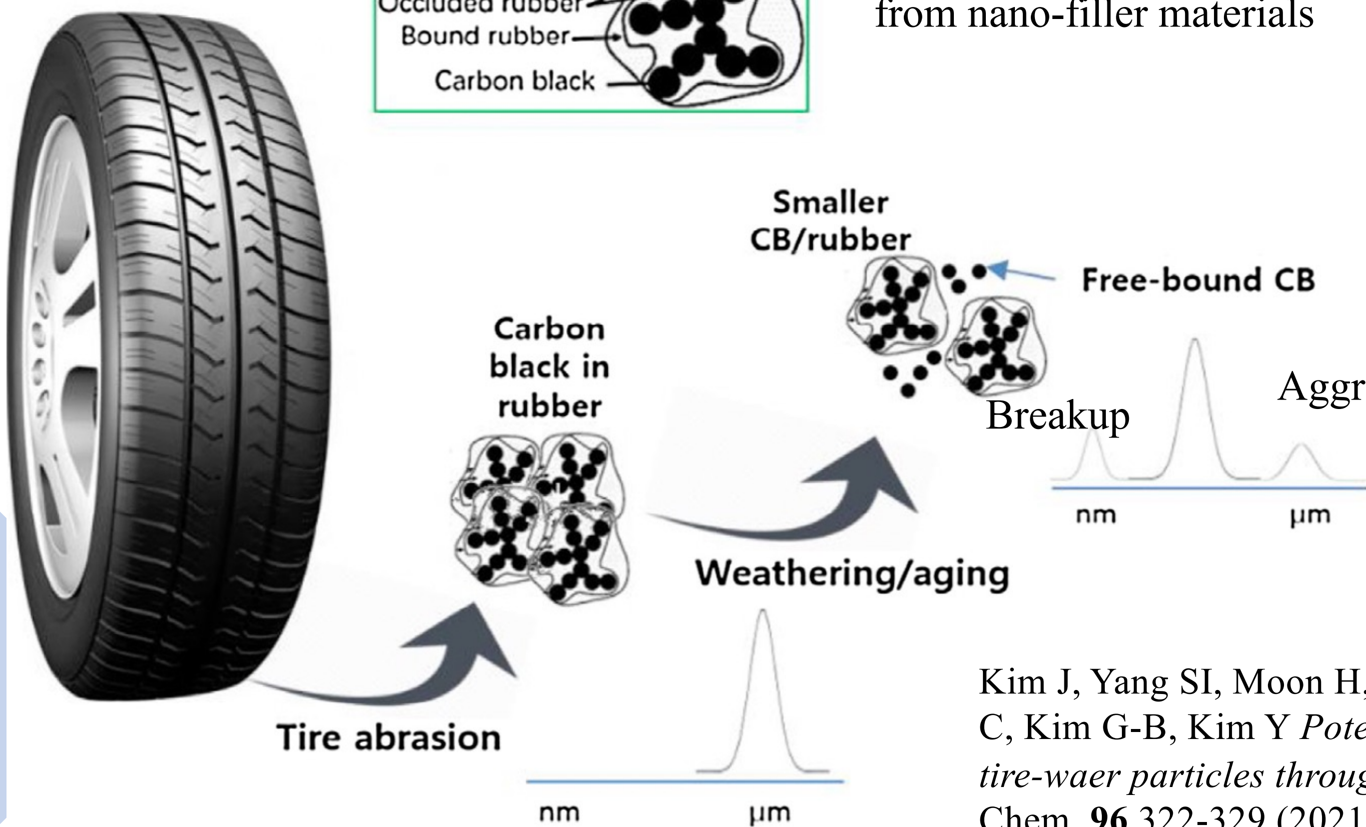
Dahl A, Gharibi A, Swietlicki E, Gudmundsson A, Bohgard M, Ljungman A, Blomqvist G, Gustafsson M *Traffic-generated emission of ultrafine particles from pavement-tire interface* Atoms. Env. **40** 1314-1323 (2006).

Greg Beaucage Univ. Cincinnati beaucag@uc.edu

# The Weathering Effect




To what extent is bound rubber separated from nano-filler materials




Aggregation/Agglomeration

Kim J, Yang SI, Moon H, Hong J, Hong J, Choi W, Son H, Lee B-C, Kim G-B, Kim Y *Potential release of nano-carbon black from tire-waer particles through the weathering effect* J. Ind. Eng. Chem. **96** 322-329 (2021).

# The Weathering Effect

Grind the surface of a passenger car tire (Nexen CP21 145R13C, Nexen tire, Korea) using a steel grater with ca. 100 mm holes.

Fragmentation of the TWP was conducted by abrasion through a mechanical process using a high-energy ball mill (planetary mill, Fritsch).

Road samples were also collected.

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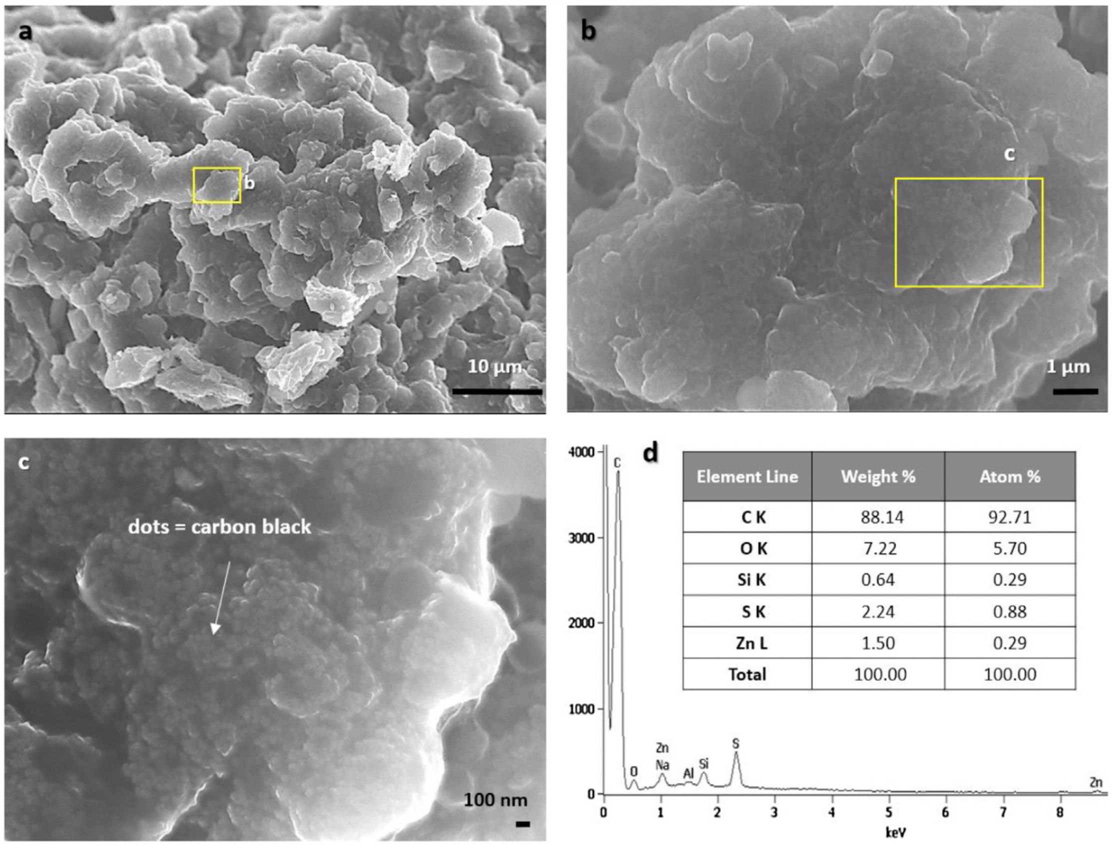
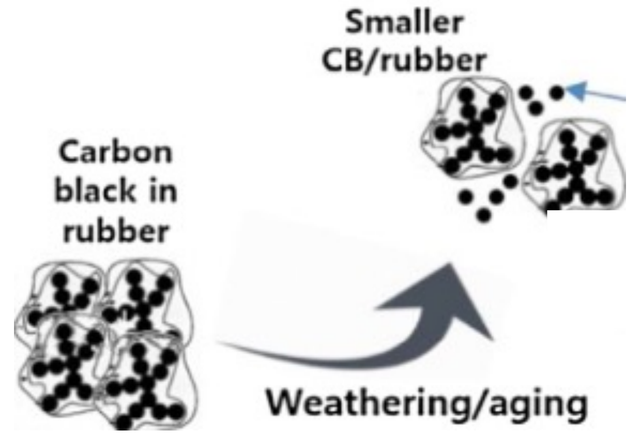



| Element Line | Weight % | Atom % |
|---|---|---|
| C K | 88.14 | 92.71 |
| O K | 7.22 | 5.70 |
| Si K | 0.64 | 0.29 |
| S K | 2.24 | 0.88 |
| Zn L | 1.50 | 0.29 |
| Total | 100.00 | 100.00 |

**Fig. 2.** SEM images and EDS results of tire-wear particles before the ball-milling test.

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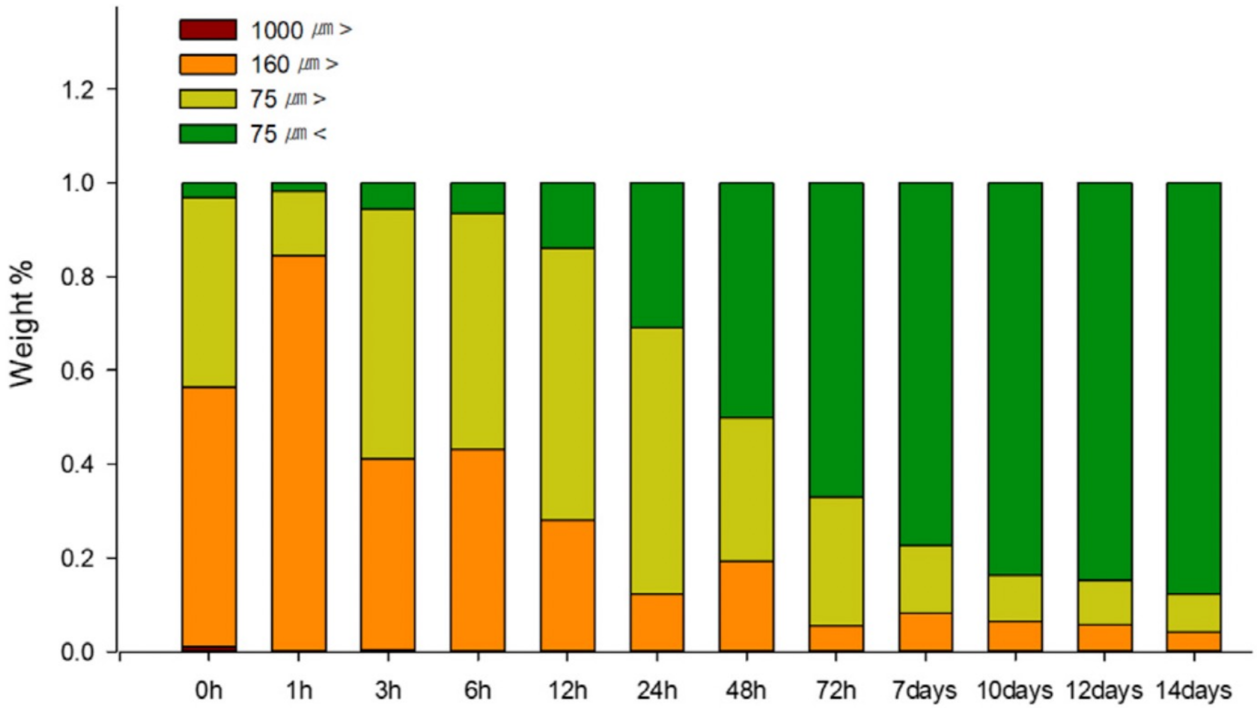


**Fig. 4.** Distribution of the weight percent of tire-wear particles with weathering time.

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# The Weathering Effect

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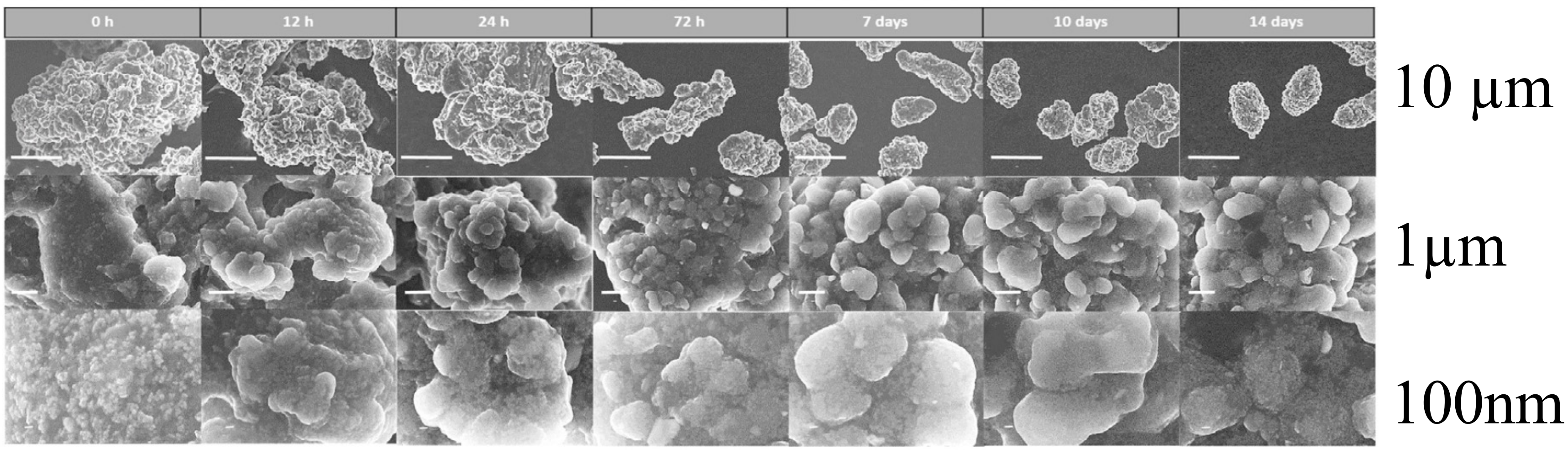



**Fig. 5.** SEM images of the tire-wear particles with weathering time. Scale bar: 10 μm, 1 μm, and 100 nm for the top, middle, and bottom images, respectively.

Kim J, Yang SI, Moon H, Hong J, Hong J, Choi W, Son H, Lee B-C, Kim G-B, Kim Y *Potential release of nano-carbon black from tire-waer particles through the weathering effect* J. Ind. Eng. Chem. **96** 322-329 (2021).

# The Weathering Effect



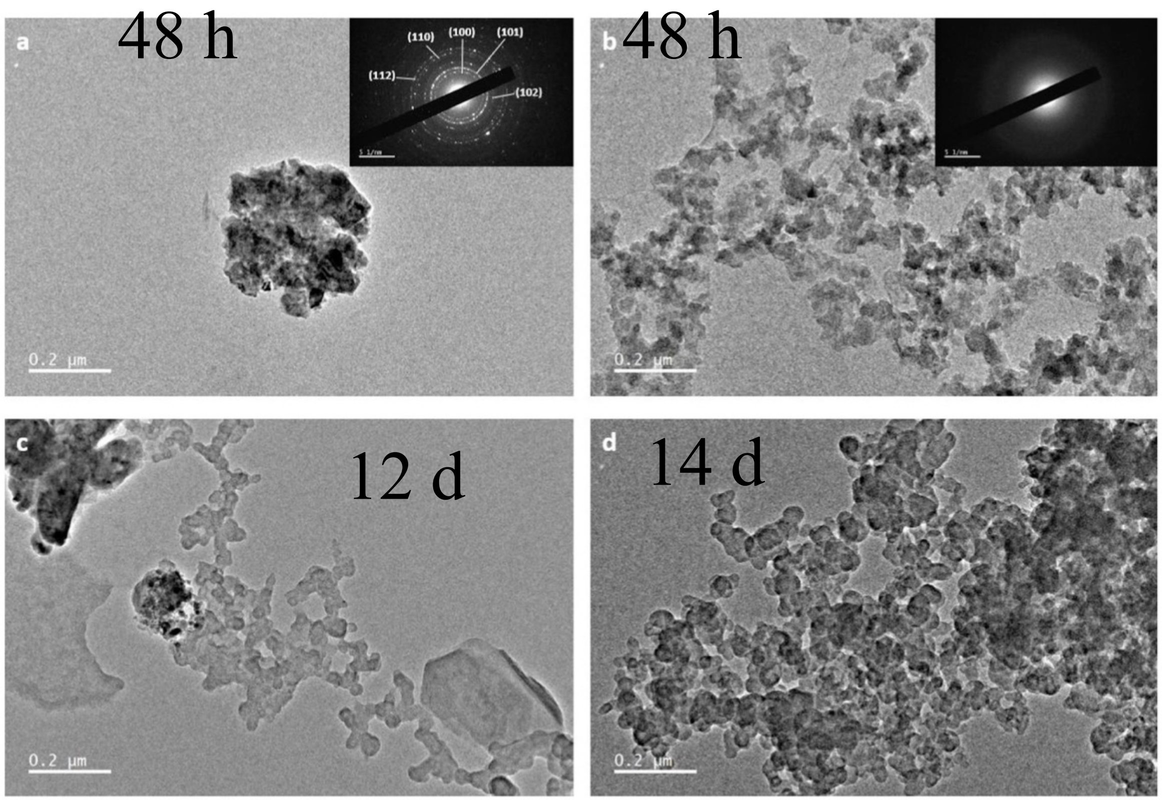



**Fig. 7.** TEM images of tire-wear particles obtained from the weathering test after (a and b) 48 h, (c) 12 days, and (d) 14 days. Inset in figures (a) and (b): ED patterns of figure (a) and (b).

Greg Beaucage Univ. Cincinnati beaucag@uc.edu

# The Weathering Effect

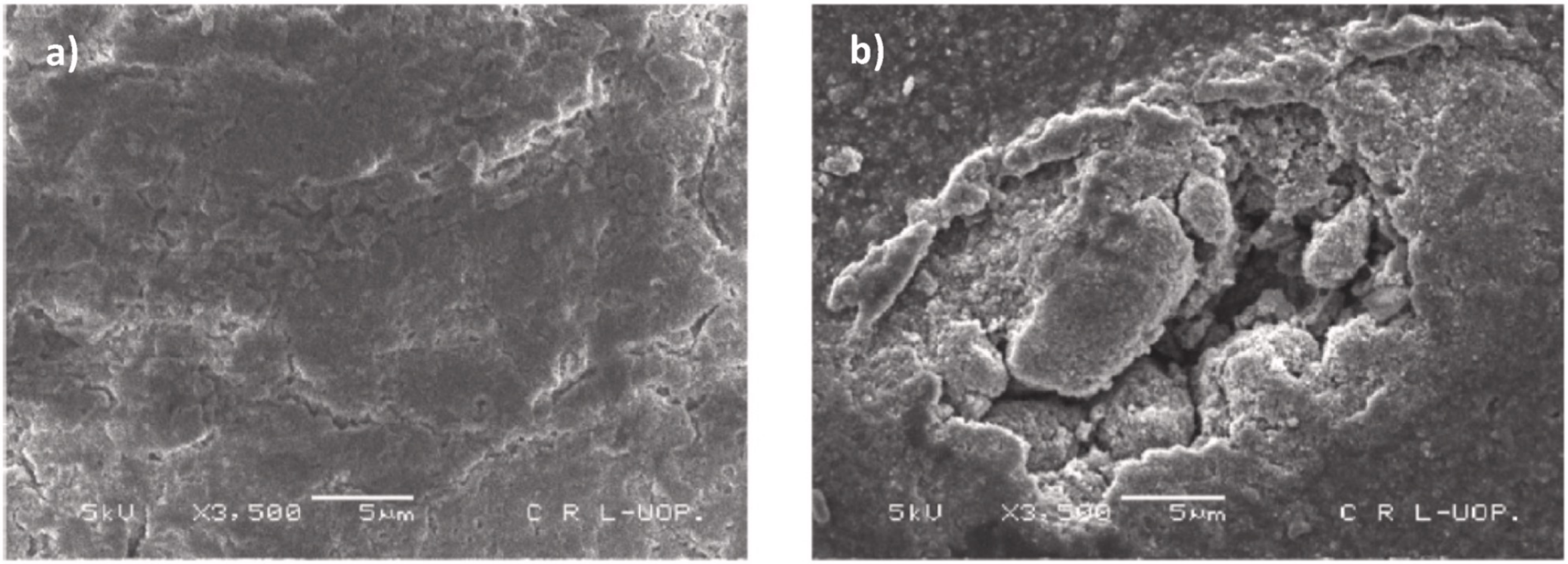



**Fig. 4.** Scanning electron micrographs of tire pieces after treatment with Bacillus sp. S-10 in mineral salt medium for 28 days at different temperatures. Rubber pieces were washed with sterilized distilled water before SEM. (a) Abiotic control; (b) surface erosion and pits formation after treatment with strain S-10 at 25 °C (Shah et al., 2013); Reprinted from International Biodeterioration & Biodegradation 83, Aamer Ali Shah, Fariha Hasan, Ziaullah Shah, Nida Kanwal, Samia Zeb, Biodegradation of natural and synthetic rubbers: A review, 145–157, 2013, with permission from Elsevier.

Wagner S, Klöckner P, Reemtsma T *Aging of tire and road wear particles in terrestrial and freshwater environments – A review on processes, testing, analysis and impact* Chemosphere **288** 132467 (2022).

# Mechanisms:

# Calculation of adhesion energy for various particles

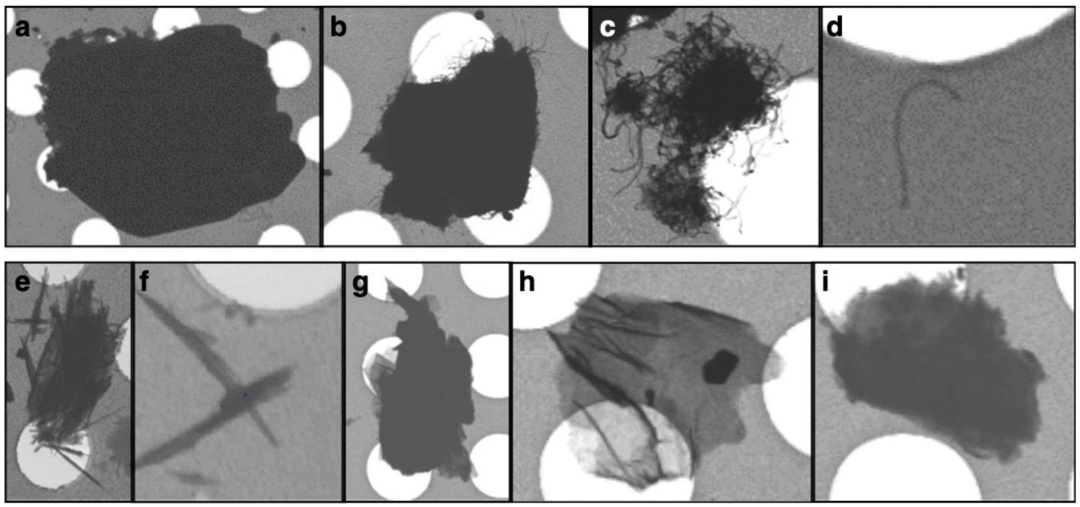
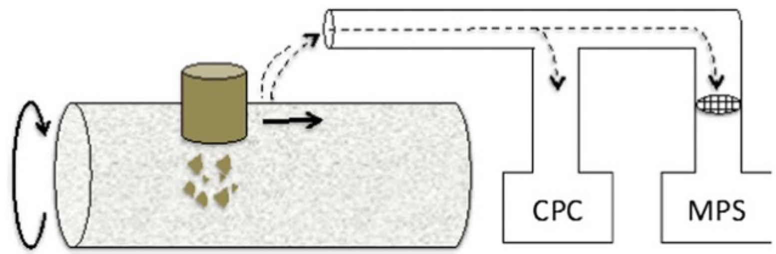
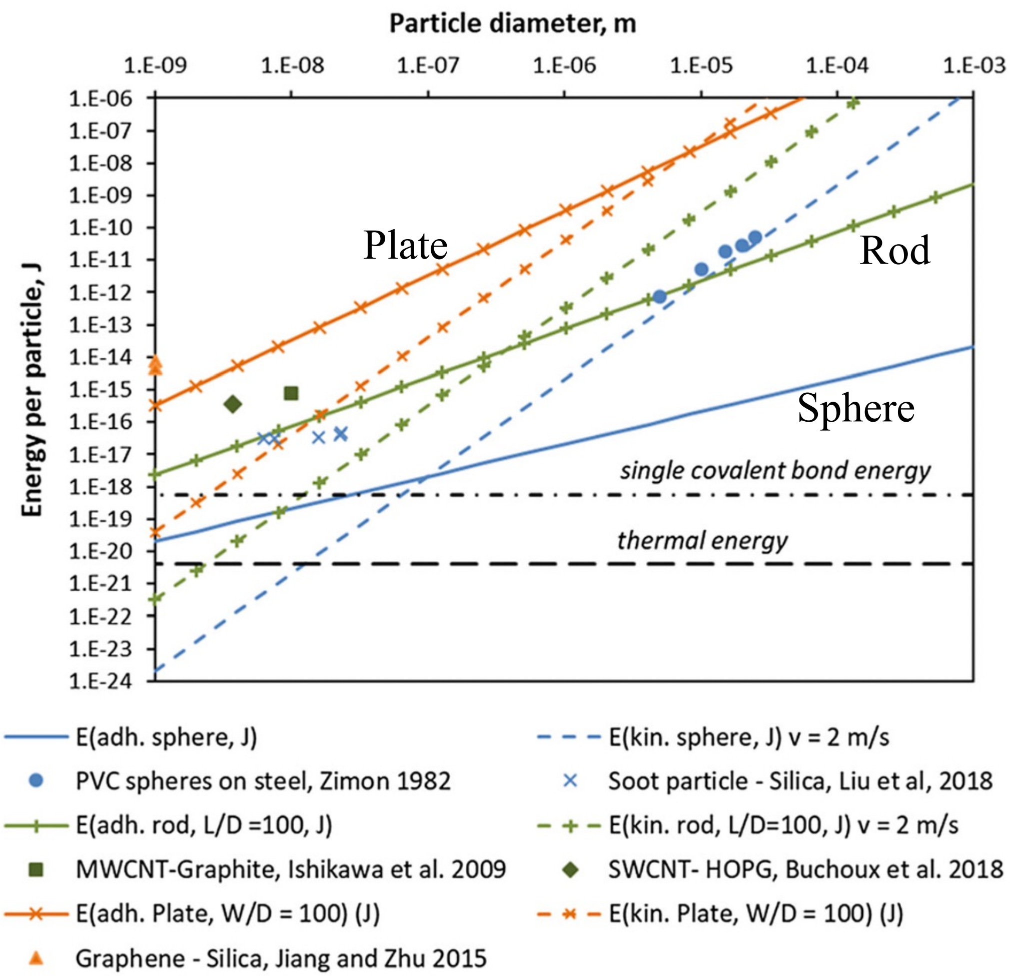


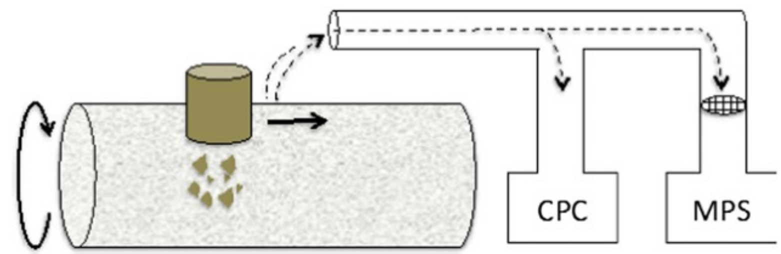

**Fig. 3** Particles captured on TEM grids during rubber abrasion: **a**, rubber particle; **b**, rubber particle with protruding MWCNT; **c**, undispersed MWCNT agglomerate; **d**, isolated MWCNT; **e**, clay agglomerate; **f**, clay particle; **g**, rubber particle with protruding RGO; **h**, isolated RGO; **i**, isolated GNP. The hole diameter is 1.5 µm




Waquier L, Myles BS, Henrard L, Vautard F Pappas CM, Feneon B, Delaitre C, Mehlem JJ, Khripin, *Quantitative measurement of nanoparticle release from rubber composites during fabrication and testing* J. Nanopart. Res. **22** 251 (2020).

# Calculation of adhesion energy for various particles




Condensation Particle Counter (CPC, Model 3007 TSI Inc.) and Mini Particle Sampler (MPS, Ecomeasure Inc.) connected to a calibrated pump (GilAir Plus, Sensidyne Inc.)

Waquier L, Myles BS, Henrard L, Vautard F Pappas CM, Feneon B, Delaitre C, Mehlem JJ, Khripin, *Quantitative measurement of nanoparticle release from rubber composites during fabrication and testing* J. Nanopart. Res. **22** 251 (2020).

# Mechanisms:

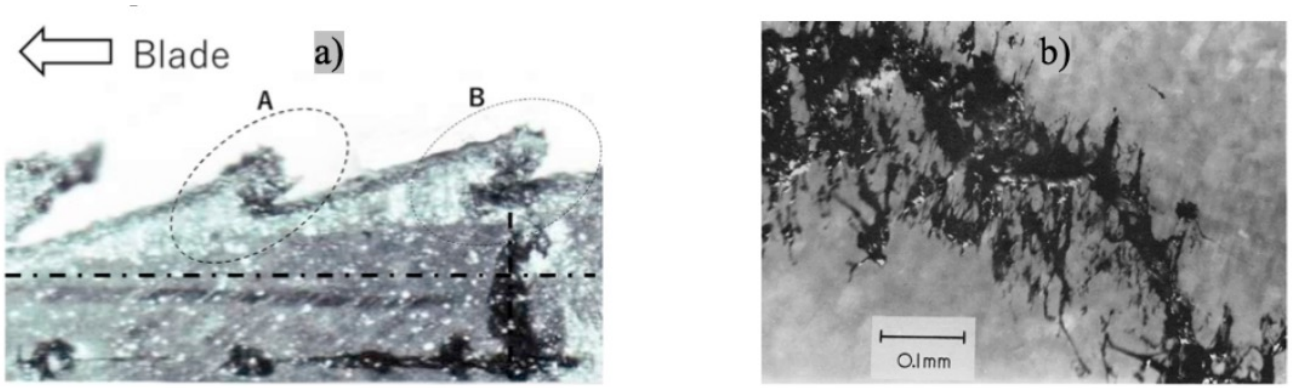
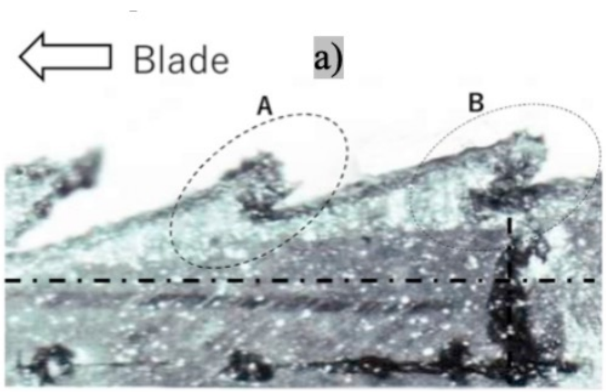


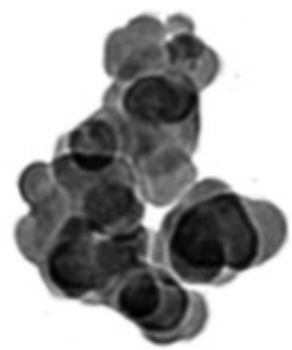

*Figure 4. a) Stick slip abrasion. [19] b) Oily film due to breakdown of polymer and blooming of oil added in the tread compound. [20]*

18) Southern E, Thomas AG *Studies of Rubber Abrasion* Rubber Chem. Tech. **52** 1008-1018 (1979).
19) Fukahori Y, Gabriel P, Liang J, Busfield JJC, *A new generalized philosophy and theory for rubber friction and wear* Wear **446-447** 203166 (2020).
20) Gent AN, Pulford CTR *Mechanisms of Rubber Abrasion* J. Appl. Polym. Sci. 28 943-960 (1983).

# Mechanisms:

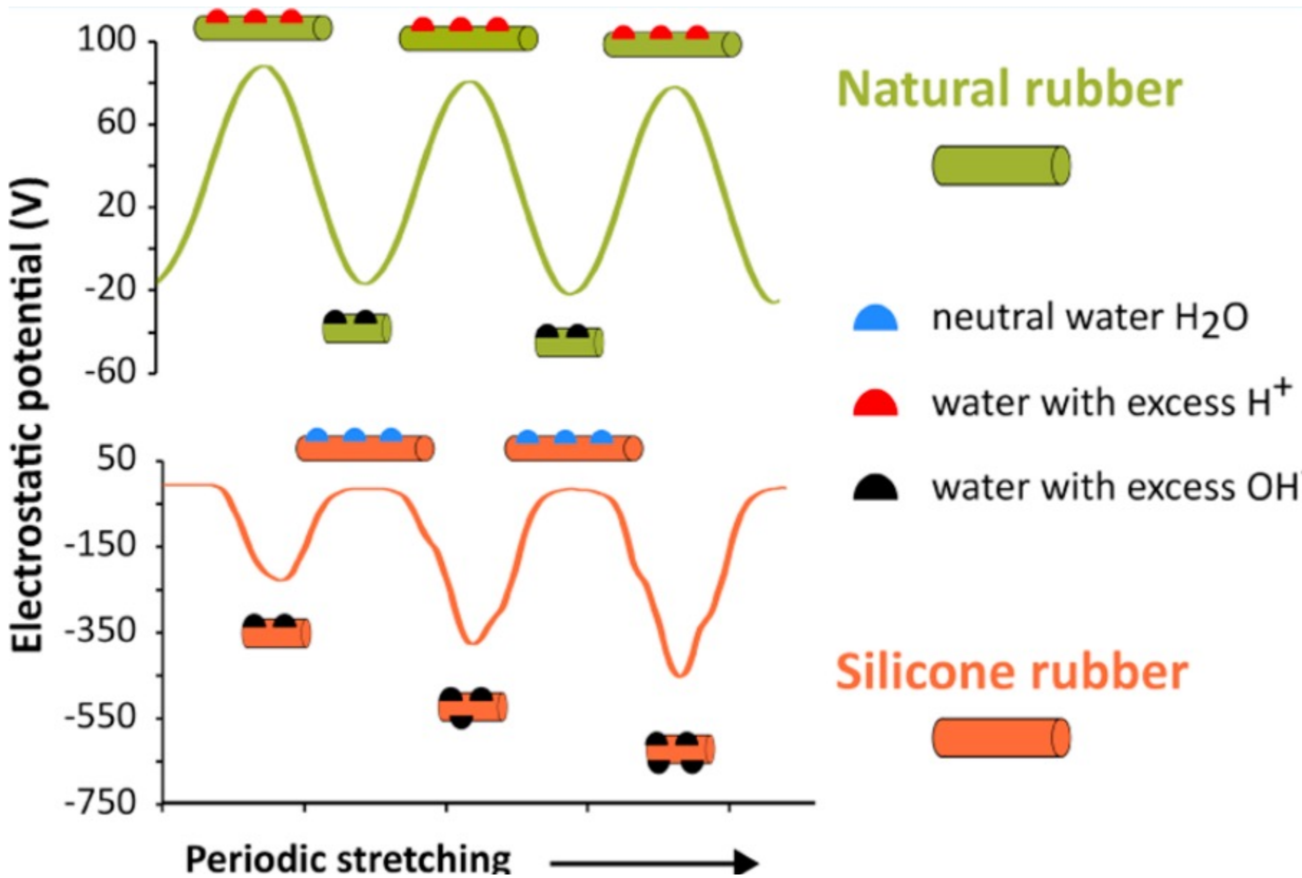



Microplastic
100 μm
95 wt % (10 no %)



Nanoparticles
100 nm
5 wt % (90 no %)

# Mechanisms:




24) Burgo TAL, Batista, BC, Galembeck F, *Electricity on Rubber Surfaces: A New Energy Conversion Effect* ACS Omega **2** 8940-8947 (2017). Campinas Brazil

# Mechanisms:

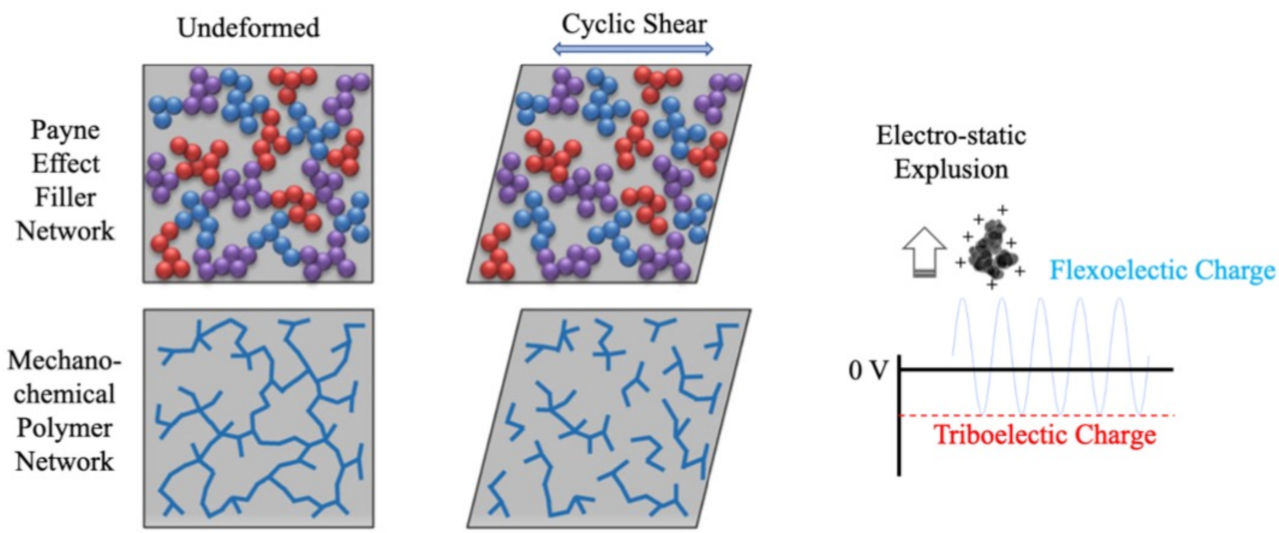



*Figure 5. Schematic of the proposed mechanism for nanoparticle release from tire tread compounds that includes breakup of the filler network, mechano-chemical degradation and breakup of the polymer network, and electrostatic repulsion of charged nanoparticles from the oppositely charged elastomer.*

# What problems need to be addressed?



Some simple questions are not answered:

- What is the mechanism for release of nanoparticles?
- Extent of occluded rubber in nascent and aged TWPs
- What is the impact of silica versus carbon black filler?
- State of static charge on TWP, and the charge distribution with particle size?
- Is such a mechanism optimized for nanoparticles (the most toxic)?
- Are there optimal tire rubber compounds (beyond wear reducing elastomers Lanxess) for minimization of TWPs or for tuning TWPs for simple removal?

# Preliminary work

Nanoparticles electrostatically deposit on the surface of tread
Particle collection using scotch tape from passenger car tires
Particle collection from the work-space and from the surface of abraded tire rubber samples
Particles were measured using:

ultra-small angle X-ray scattering, XRD, APS, SMPS, TEM, SEM, TSI Dust Trak

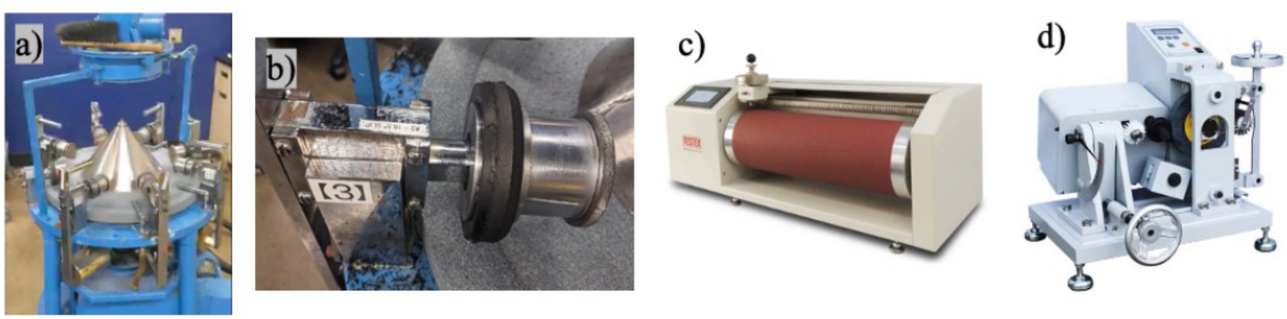



*Figure 6. Instruments to be used in this study. a) Angle abrader, Akron Rubber Development Laboratory (ARDL). b) Angle Abrader wheel. c.) Rotary Drum Abrader (ARDL). d) Portable Akron Abrader.*

# Preliminary work

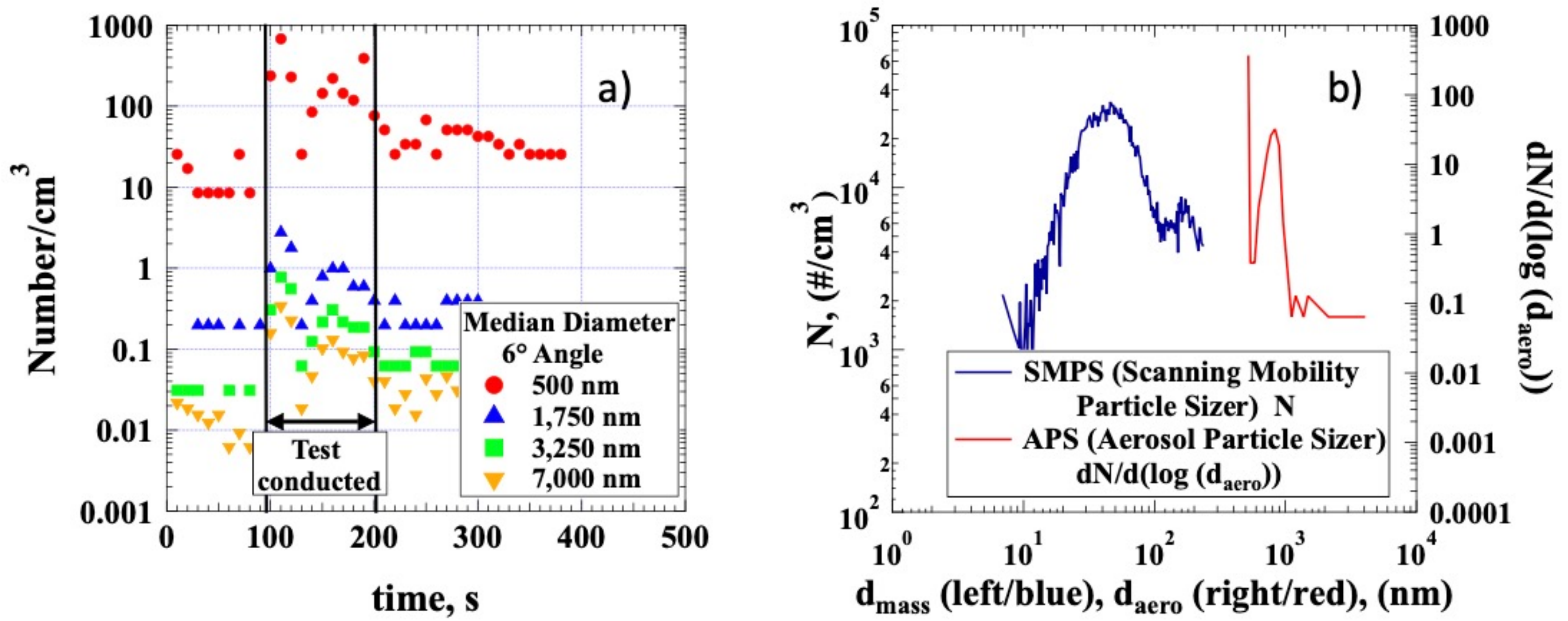
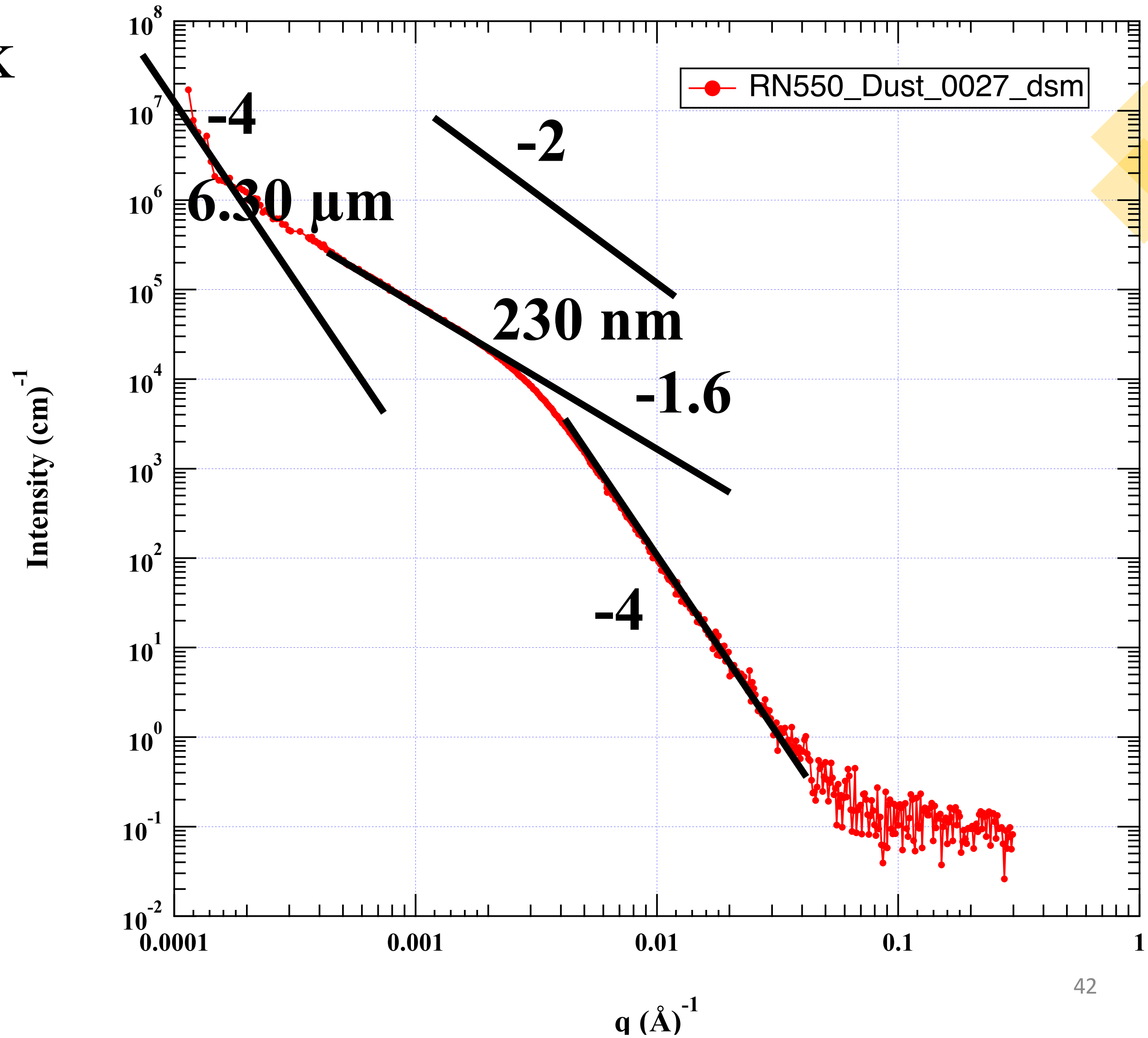


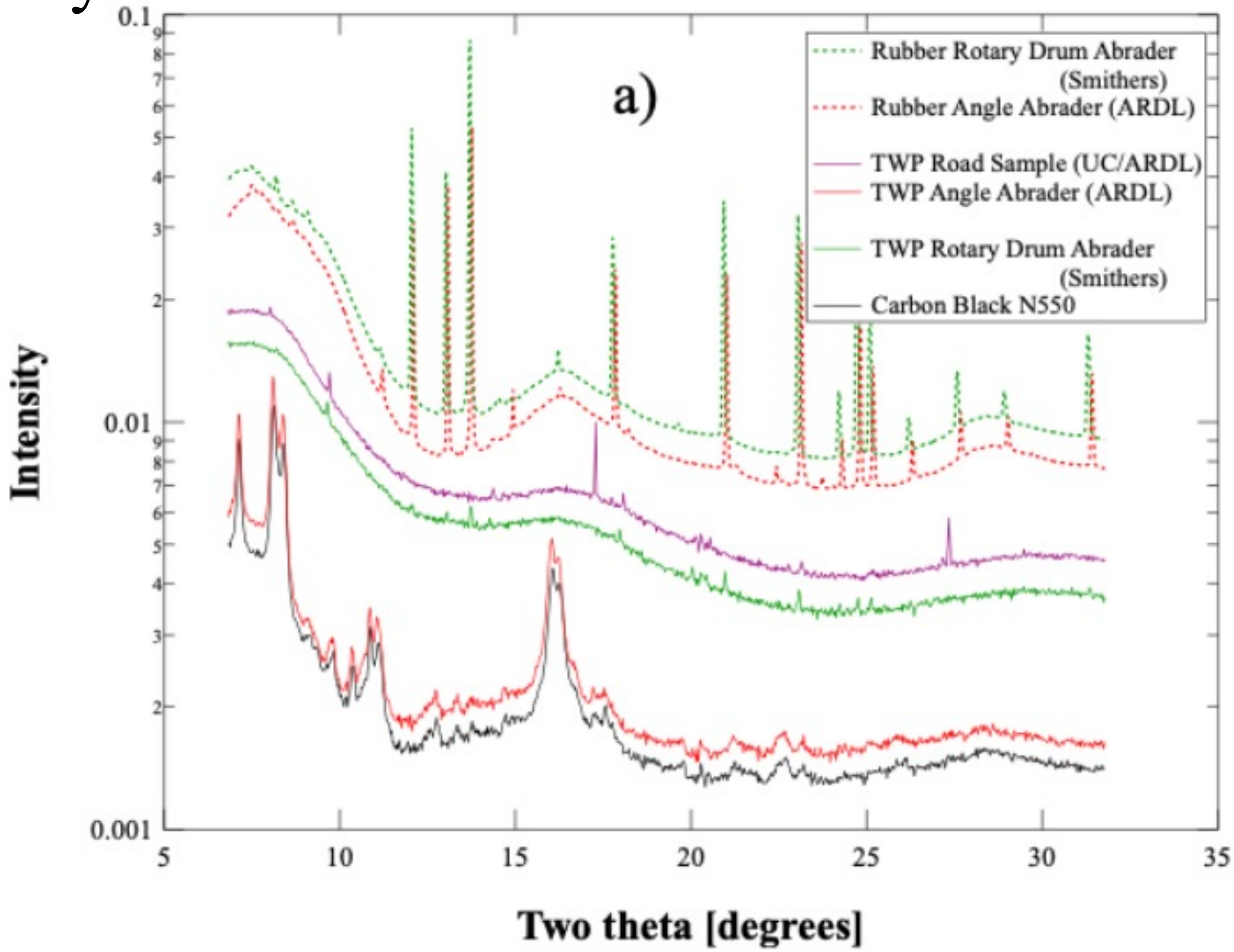

*Figure 8. a) Preliminary in situ aerosol tests using a TSI DustTrak™ II Aerosol Monitor for the angle abrader (ARDL) during wheel abrasion for a 6° wheel angle, with a maximum nano-TWP emission at about 1,000 particles/cm$^3$. b) Electrostatically collected particles from angle abrader (ARDL) measured at HELD.*

# Preliminary Work

Carbon
Black N550

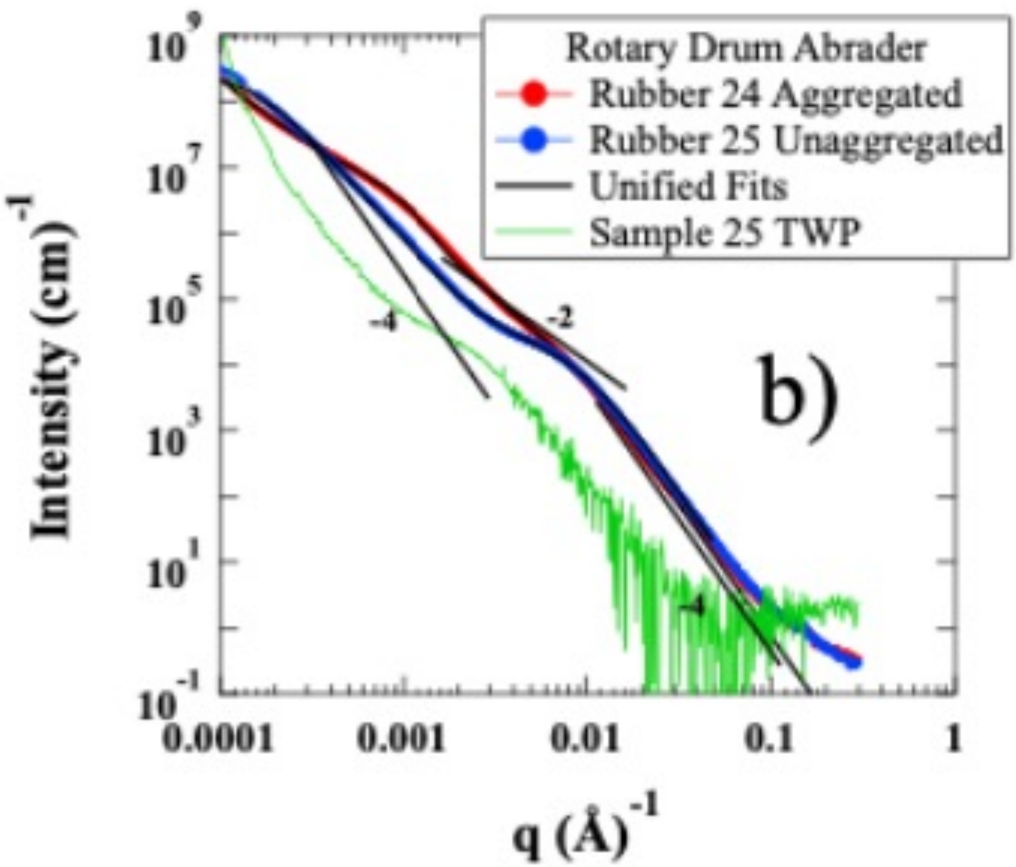


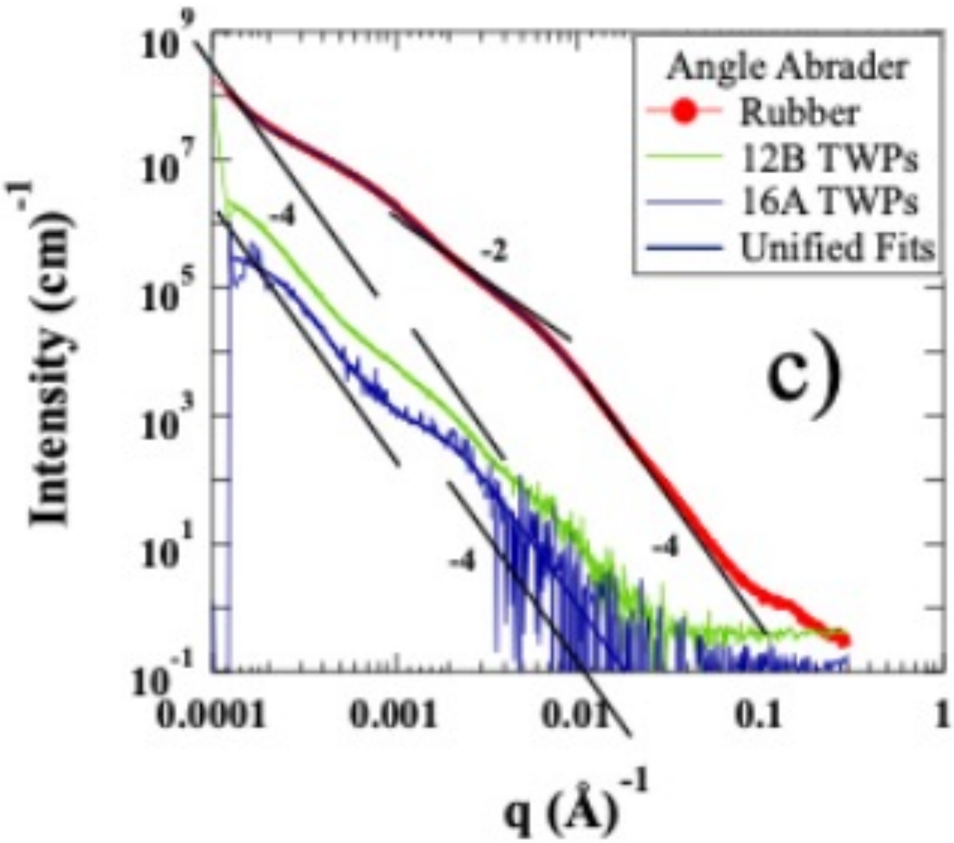
# Preliminary work



Greg Beaucage Univ. Cincinnati beaucag@uc.edu

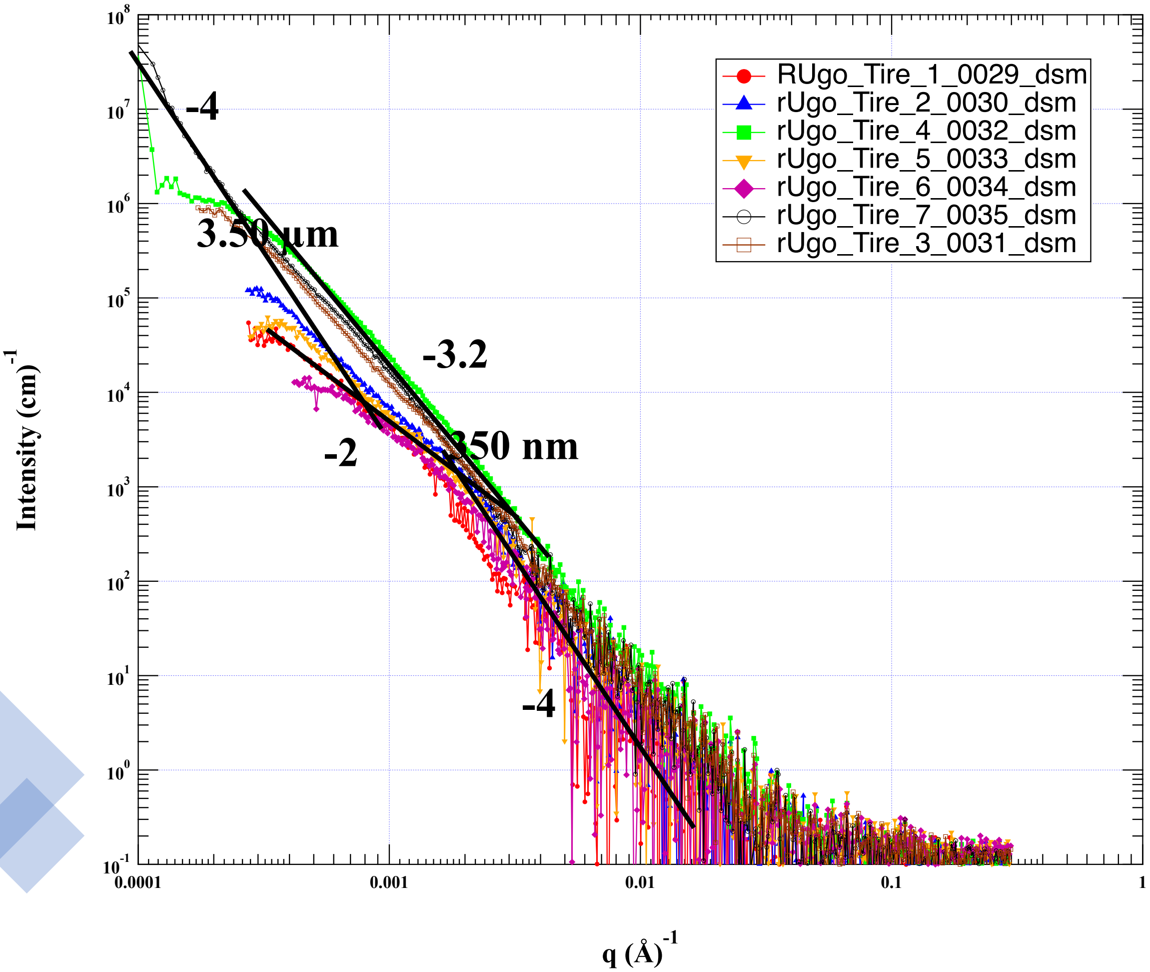
# Preliminary work



Greg Beaucage Univ. Cincinnati beaucag@uc.edu

# Preliminary Work




## Tire Dust From Actively Used Consumer Tires

- Some samples show micron size agglomerates (3, 4, 7)
- Some show nano-size aggregates (2, 5, 6)

# Preliminary work

**Table 1 Fit results from USAXS measurements.**

| Sample | $d_p$, nm | $d_f$ | c | $d_{min}$ | DOA, z | Aggregate $R_g$, nm | Network $d_f$ |
|---|---|---|---|---|---|---|---|
| Car TWP | 142 | 2.4 | 1.1 | 2.2 | 65 | 641 | – |
| Angle Abrader Rubber | 32.5 | 2.6 | - | - | 549 | 478 | – |
| A. A. TWP | Three unaggregated particles: $d_{p1}$ = 33.3 nm; $d_{p2}$ = 161 nm; $d_{p3}$ = 1580 nm | | | | | | – |
| Rotary Drum Abrader Rubber | 24.7 | 2.6 | 1.5 | 1.8 | 421 | 209 | 2.26 |
| R. D. TWP | 52.3 | 2.7 | - | - | - | 224 | - |

# Preliminary work

**Table 1 Fit results from USAXS measurements.**

| Sample | $d_p$, nm | $d_f$ | c | $d_{min}$ | DOA, z | Aggregate $R_g$, nm | Network $d_f$ |
|---|---|---|---|---|---|---|---|
| Car TWP | 142 | 2.4 | 1.1 | 2.2 | 65 | 641 | – |
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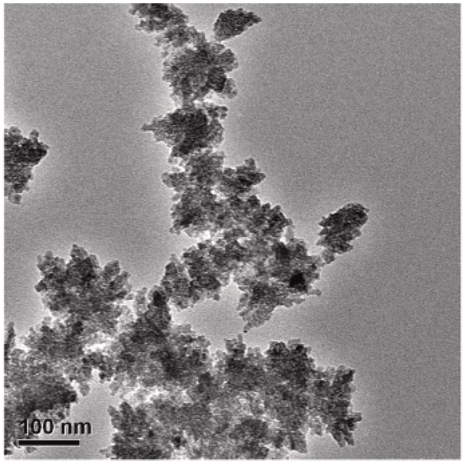



Li Y, Shi T, Li X, Sun H, Xia X, Ji X, Zhang J, Liu M, Lin Y, Zhang R, Zheng Y, Tang J *Inhaled tire-wear microplastic particles induced pulmonary fibrotic injury via epithelial cytoskeleton rearrangement* Env. Int. **164** 107257 (2022).

Karlsruhe Instrument

# Preliminary work

a) b) c)

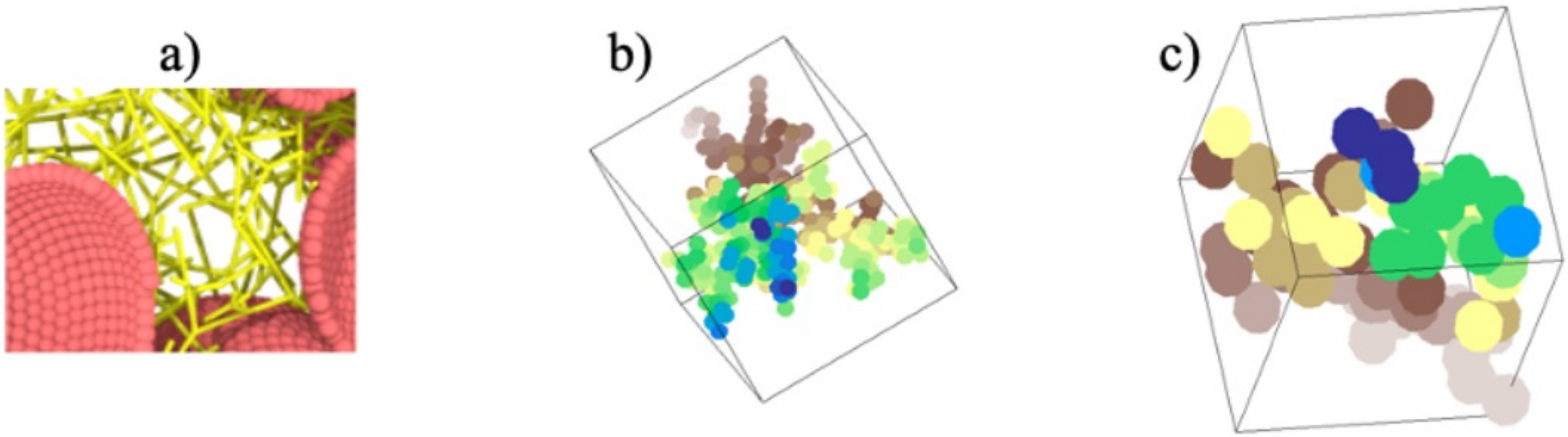
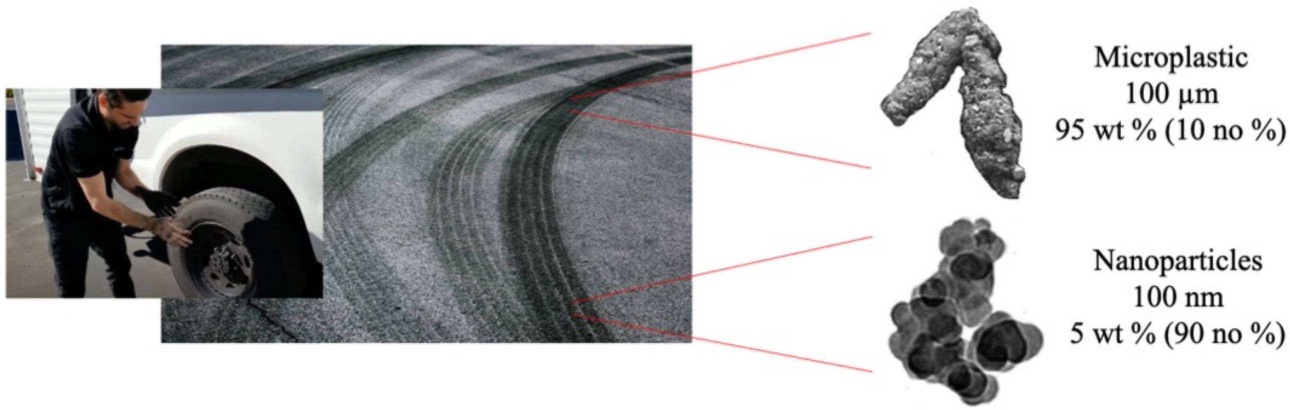


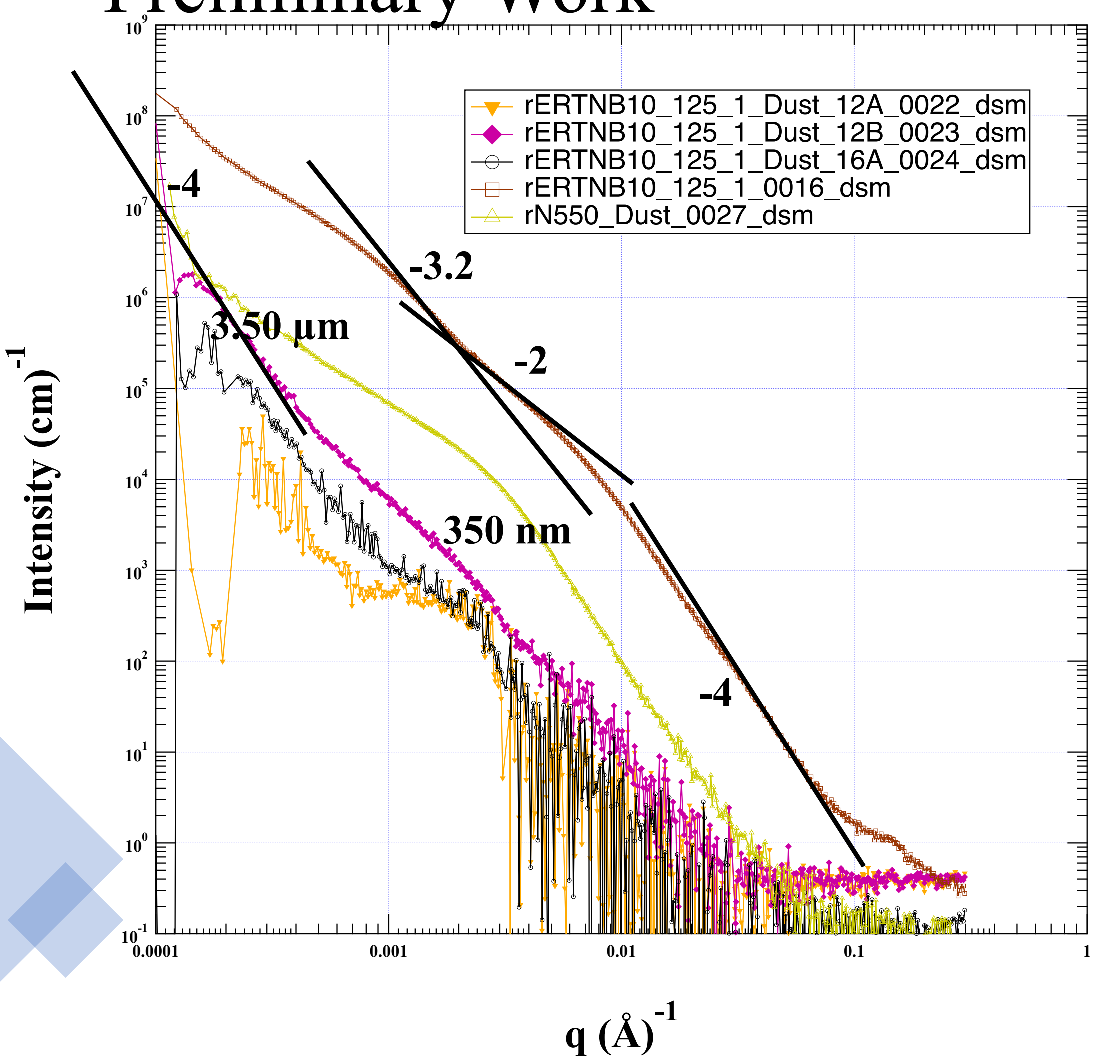
*Figure 10. a) Spherical shell nanoparticles of about 0.5 μm diameter from Lin et al [47] using LAMMPS. b) Particles from the rotary drum abrader using the Rubber 24 sample from Table 1 and Figure 9b. c) TWPs from car sample in Table 1. (Figures b and c are 3D aggregated nanoparticle models from USAXS analyses using the method of Mulderig et al. [55,58].)*

# Summary




- How large of a problem are nano-tire wear particles (nTWPs)?
- Preliminary data indicates significant number density of nanoparticles.
- These particles seem to be electrostatically adhering to the tire in some cases which may be selective for small particles.
- The nanoparticles may serve as vectors for various chemicals in the tire such as thiols, 6PPD (Washington State study)

# Preliminary Work




Comparison of Tire Compound/Dust in the Lab Near Abrader/N550 CB

- Significant differences
- Lab dust is larger and simpler, mostly CB
- Lab dust has variability
- Compound has at least two structural fillers