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Long-term aging and degradation of microplastic particles: Comparing in situ oceanic and experimental weathering patterns

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ABSTRACT

Polypropylene, low-density polyethylene, and high-density polyethylene pre-production plastic pellets were weathered for three years in three experimental treatments: dry/sunlight, seawater/sunlight, and seawater/darkness. Changes in chemical bond structures (hydroxyl, carbonyl groups and carbon-oxygen) with weathering were measured via Fourier Transform Infrared (FTIR) spectroscopy. These indices from experimentally weathered particles were compared to microplastic particles collected from oceanic surface waters in the California Current, the North Pacific Subtropical Gyre, and the transition region between the two, in order to estimate the exposure time of the oceanic plastics. Although chemical bonds exhibited some nonlinear changes with environmental exposure, they can potentially approximate the weathering time of some plastics, especially high-density polyethylene. The majority of the North Pacific Subtropical Gyre polyethylene particles we measured have inferred exposure times > 18 months, with some > 30 months. Inferred particle weathering times are consistent with ocean circulation models suggesting a long residence time in the open ocean.

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1. Introduction

Plastics in the ocean, particularly in the North Pacific Subtropical Gyre, have been of concern for decades (Carpenter and Smith, 1972; Wong et al., 1974). Recent studies estimate that there may be approximately five trillion pieces of plastic in the global ocean, with an estimated 4.8 to 12.7 million metric tons entering the ocean annually (Eriksen et al., 2014; Jambeck et al., 2015). Eriksen et al. (2014), along with others (Hidalgo-Ruz et al., 2012; Goldstein et al., 2013), state that the vast numerical majority of plastics in the ocean are microplastic, or particles < 5 mm in diameter. However, there is currently no method that estimates how long a given microplastic particle has been in the ocean. The small size of fragmented, weathered particles also makes it impossible to trace these particles to their source (Jambeck et al., 2015). Knowing how long a particle has been in the ocean is critical for calculating the residence time of particles in different regions of the ocean, testing the accuracy of models, and assessing the efficacy of marine debris mitigation policy.

The small fragments of microplastic created by weathering are detrimental to ocean ecosystems for multiple reasons. Studies have shown gooseneck barnacles (Goldstein and Goodwin, 2013), mesopelagic fishes (Davison and Asch, 2011), Norway lobsters (Murray and Cowie, 2011), and other small animals can consume microplastics *in situ*, and other invertebrates have been shown to eat them in lab settings (Murray and Cowie, 2011; Cole et al., 2013; Wright et al., 2013).

Synthetic microfibers and microplastics are small enough to physically accumulate and to translocate from an organism's gut into its circulatory system (Browne et al., 2008). Some plastics contain harmful chemical additives (e.g. PCBs – polychlorinated biphenyls) that can bioaccumulate in marine organisms, leading to liver toxicity and other deleterious physiological effects (Rochman et al., 2013). Since plastics' hydrophobicity causes them to sorb marine and atmospheric persistent organic pollutants, there is also concern for bioaccumulation of these pollutants from plastic ingestion (Ogata et al., 2009).

Microplastics are currently impossible to remove *en masse* from the open ocean due to their small size, chemical inertness, similar dimension and distribution as plankton and fish eggs, and their distribution over the vast extent of the oceanic gyres. Thus, it is essential to understand processes that lead to the accumulation and degradation of plastic particles, as well as to develop strategies to limit inputs into the ocean (Jambeck et al., 2015).

Although many studies have examined aging of polyethylene and polypropylene (Stark and Matuana, 2004; La Mantia and Morreale, 2008), almost all have been conducted in accelerated weathering devices that use much higher temperatures than natural weathering (Stark and Matuana, 2004). Elevated temperatures can lead to different chemical reactions than those that occur naturally (Lacoste and Carlsson, 1992; Tidjani, 2000).

There have been some studies of the natural weathering of plastics: Andrady et al. (1993) examined natural weathering of LDPE, and Rajakumar et al. (2009) examined natural weathering of PP; both experiments tested sheets of plastic film in ambient air and rain. Andrady (1990) compared the weathering of LDPE films in ambient

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air and ambient seawater. Pegram and Andrady (1989) tested LDPE film, PP strapping tape, latex balloons and trawl netting in both ambient air and seawater. Though these studies are very useful, the numerical majority of marine debris is not intact films or objects but rather microplastic particles (Goldstein et al., 2013). Also, most previous studies extend for a maximum of three months, with some for only a few weeks (Lacoste and Carlsson, 1992; Andrady et al., 1993; La Mantia and Morreale, 2008), although Andrady (1990) and Pegram and Andrady (1989) weathered samples for a year. There is a need for greater understanding of the longer term, natural weathering of microplastics and the variables that interact in that weathering process (Tidjani, 2000). In addition, knowing how microplastic particles weather is important for understanding the ecological impacts of the most common type of marine debris.

The present study's unique results stem from longer term (i.e., 3 year) controlled exposure to natural sunlight and ambient seawater. It is therefore a more realistic proxy for the weathering processes that plastic particles experience in the open ocean than many previous studies. This is also the first study to directly compare naturally weathered plastic particles to particles collected from the ocean in an attempt to quantify the exposure time of the oceanic particles.

2. Materials and methods

2.1. Weathering experiment

Beginning in December 2010, preproduction pellets (or nurdles) of the six most common consumer plastics (Andrady, 2003) were exposed to three treatments: dry/sunlight, seawater/darkness, or seawater/sunlight, in comparison with dry/darkness control treatments. The dry/sunlight treatment roughly approximates the weathering conditions of dried plastic particles on beaches; seawater/darkness simulates conditions similar to those found in some benthic environments; seawater/sunlight simulates exposure of particles floating at the air-sea interface. The six consumer plastics were polyethylene terephthalate (PET; Resin ID #1), high density polyethylene (HDPE; Resin ID #2), polyvinyl chloride (PVC; Resin ID #3), low density polyethylene (LDPE; Resin ID #4), polypropylene (PP; Resin ID #5), and polystyrene (PS; Resin ID #6) (American Chemistry Council, 2010).

For the dry/sunlight treatment, 250 mL of each type of preproduction pellet were placed in Pyrex glass trays on the roof of Hubbs Hall at Scripps Institution of Oceanography, La Jolla, California, 32.867°N, 117.257°W. Each tray was covered by fiberglass screening (2 mm mesh size) to prevent pellet loss. Each type of plastic was placed in two trays on the roof (N = 2), except for PVC (N = 1 due to a shortage of supply pellets). The roof was covered in naturally colored pebbles, having a similar albedo effect as most beaches, and had unoccluded natural sunlight throughout daylight hours year-round.

For the seawater/darkness and seawater/sunlight treatments, 250 mL of each type of preproduction pellet were placed in 75.7 L (20 gal) aquaria with flowing seawater (N = 2 for each treatment, N = 1 for PVC). To separate plastic types, aquarium divider screens were installed. Each plastic type was randomly assigned to a location in the tank, with different locations in the two replicate tanks. Local seawater from the Scripps running seawater system (intake from the seaward end of the Scripps Pier) flowed continuously through a sprinkle bar placed over the tank, and drained through a screen-covered standpipe. The seawater/darkness treatment tanks were placed in an indoor experimental room and covered in opaque black plastic sheeting, which was only removed when the tanks were sampled. The seawater/sunlight tanks were placed side-by-side with the dry/sunlight treatments, and the tops of the aquaria were covered with fiberglass screening to prevent pellet loss.

From December 2010 to July 2012, the experiment was sampled monthly by removing ten pellets from each replicate. After July 2012, the tanks were all cleaned monthly, but the pellets were sampled bi-monthly. After removal, pellets were gently wiped to remove epiphytes,

with deionized water, dried at 60 °C for 24 h, and stored in glass vials in the dark at room temperature until Fourier-Transform Infrared Spectrometer (FTIR) analysis.

Seven time points were selected for analysis: T_0 = unweathered particles, T_5 = 5 months of weathering, T_9 = 9 months, T_{13} = 13 months, T_{18} = 18 months, T_{30} = 30 months, and T_{36} = 36 months. Only HDPE, LDPE and PP were analyzed for the experimental study because they are the most common plastics found at the ocean's surface, due to their common commercial use and positive buoyancy (Freund Container & Supply, 2010). In 2012, PE and PP accounted for 63% of the plastic waste in the United States (EPA, 2014).

2.2. Oceanic samples

In August 2009, samples were collected on the Scripps Environmental Accumulation of Plastic Expedition (SEAPLEX) cruise on the R/V *New Horizon* (Fig. 1). Samples were collected using a standard Manta net (0.86 m wide × 0.2 m high mouth opening) (Brown and Cheng, 1981) with 333 μm mesh, towed for 15 min at 0.7–1 m s⁻¹. Water volume flowing through the net was measured with a calibrated General Oceanics analog flowmeter. Samples were fixed in 1.8% formaldehyde buffered with sodium tetraborate.

Each sample was sorted for microplastic at 6–12× magnification under a Wild M-5 dissecting microscope. Plastic particles were removed, dried at 60 °C, and stored in glass vials in the dark at room temperature. If there were fewer than 50 particles per sample, the entire sample was analyzed. If there were >50 particles per sample, the sample was split using the quartering method (ASTM Standard C702/C702M-11, 2011) until an aliquot of 30–50 particles was obtained. Particles were then soaked for 12 h in 10% hydrochloric acid to remove calcium carbonate deposits, rinsed in deionized water, re-dried at 60 °C, and stored in glass vials in the dark at room temperature.

For the present study analysis, the California Current was defined as having a surface temperature < 19 °C and surface salinity < 33.5 (Lynn and Simpson, 1987). The North Pacific Subtropical Gyre (NPSG) was defined as having surface temperatures > 22 °C and salinity > 34.8 (Rodén, 1980; Niiler and Reynolds, 1984). The transition region was defined as having a surface temperature of 19–22 °C and surface salinity of 33.5–34.8 (Rodén, 1980; Lynn and Simpson, 1987). Because only surface data were used, these should be viewed as approximations rather than absolute oceanographic definitions (Goldstein et al., 2013). Fig. 1 reflects the sampling locations of the SEAPLEX cruise, with filled shapes indicating those sampling stations analyzed using FTIR in this study. These stations were chosen so that they were distributed throughout the cruise track, without reference to the abundance of plastic in each sample. Median bond indices (see below) from the three regions were

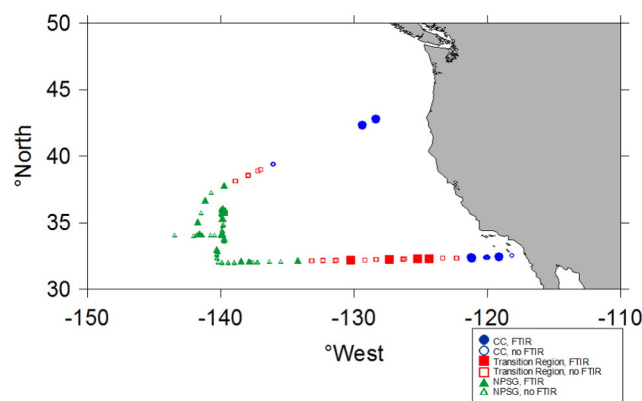


Fig. 1. SEAPLEX Manta net sampling locations. California Current (blue circles), transition region (red squares) North Pacific Subtropical Gyre (green triangles). Points indicate all locations sampled via Manta net; solid symbols are Manta samples analyzed by FTIR for this study.

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