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Electrical Percolation Controlled by Filler Migration in PS/PPE/SWCNT Blend Composites

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Abstract:
Polystyrene (PS) and poly(2,6-dimethyl-1,4-phenylene ether) (PPE, also called PPO) form one of the few commercially important miscible polymer pairs, and this fact shapes any discussion of nanotube migration in PS/PPE composites. This paper compares published evidence on PS/PPE phase behaviour with the literature on selective localization of carbon nanotubes in immiscible blends. Calorimetric data show a single composition dependent glass transition for PS/PPE, although early dynamic mechanical work found two relaxation peaks, so the miscibility question depends on the length scale probed. In a truly single phase melt, "migration" can only mean kinetic redistribution of nanotubes during mixing; in immiscible pairs such as PC/SAN, PP/PS and PEO/PE, nanotubes cross interfaces within minutes and the threshold falls by double percolation. The one verified PPE/PS nanotube study reports a percolation threshold of 0.4 wt% multiwalled nanotubes and a conductivity rise of about twelve orders of magnitude at 0.5 wt%. Interphase migration in PS/PPE is, on this evidence, unproven; what a formulator can actually adjust is local viscosity, mixing time and the choice of a third polymer phase.

Keywords: polystyrene, poly(phenylene ether), carbon nanotubes, percolation threshold, selective localization, melt mixing

1. Introduction

Conductive polymer composites are made by dispersing a conductive filler in an insulating polymer until the particles touch, or nearly touch, across the sample. For carbon nanotubes the loading needed is small because of their large aspect ratio (length over diameter). When the matrix is a blend of two polymers a second lever appears. If the nanotubes sit in one phase only, or at the interface, the effective filler concentration in the conductive pathway rises and the threshold falls. It was first worked out for carbon black over thirty years ago [1].

PS/PPE blends occupy an odd position here. PPE is an amorphous engineering thermoplastic with a glass transition near 212 to 222 °C, too viscous to process alone, so it is sold blended with PS under trade names such as Noryl. Its repeat unit (Figure 1) is a phenylene ring with two methyl groups, linked through an ether oxygen; the aromatic backbone makes it stiff, and the lack of polar side groups lets it mix with the equally nonpolar PS.

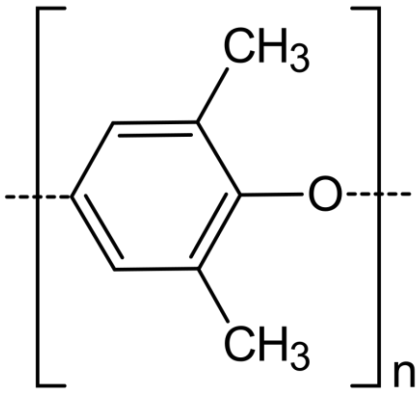


Figure 1. Repeat unit of poly(2,6-dimethyl-1,4-phenylene ether), the PPE component of the blend. Image: Dschanz, Wikimedia Commons, public domain, https://commons.wikimedia.org/wiki/File:Polyphenylenether.svg

The classic calorimetric work found a single glass transition moving smoothly between that of PS and that of PPE [2], the usual criterion for a miscible pair. If no second phase exists, nanotubes have nowhere to migrate to. But the literature is less tidy than the textbook summary. Dynamic mechanical measurements on the same blends showed two relaxation peaks, one PS rich and one PPE rich [3]; apparent compatibility, the authors concluded, depends on the size of the molecular motion the experiment samples.

This paper therefore treats the title as a question. We review what is established about PS/PPE phase behaviour, set out the percolation and wetting relations for filler networks in blends, and compare localization data from immiscible PS based blends with the few data for PPE/PS itself. Where sources used multiwalled rather than singlewalled nanotubes, this is said explicitly.

2. Scientific Background and Related Research

Miscibility of PS and PPE was examined in the early 1970s by two methods. Stoelting, Karasz and MacKnight prepared powder mixed blends with PPE weight fractions of 0.25, 0.50 and 0.75 [3]. Differential scanning calorimetry (DSC) showed one glass transition per blend; dynamic mechanical spectra showed two loss peaks, a PS rich and a PPE rich phase. Shultz and Gendron measured nine solution cast blends by DSC and found transition temperatures rising monotonically from 99 °C (PS) to 212 °C (PPE) [2]; the films were homogeneous. Cowie and co-workers later modelled structural relaxation in PS/PPE as a miscible system across the composition range [4]. So the pair is miscible, with the caveat that incomplete mixing leaves composition gradients that mechanical relaxation picks up.

Nanotube studies on PPE/PS are rare. The one verified paper is by Sathyanarayana and colleagues, who compounded 4 wt% multiwalled carbon nanotubes (MWCNT) into a miscible PPE/PS blend on a twin screw extruder and diluted the masterbatch at 400 and 600 rpm [5]. Microscopy showed good dispersion even at 4 wt%, with slightly larger agglomerates at 600 rpm. Percolation occurred at 0.4 wt% MWCNT, and conductivity at 0.5 wt% was close to twelve orders of magnitude above the unfilled blend. Nothing in the paper suggests two polymer phases.

A second line of evidence places PS/PPE inside a three polymer system. Lee and co-workers found that in PA66/(PS/PPE)/CNT the nanotubes sit mainly in the polyamide, and nanotubes first placed in PS/PPE

moved into PA66 as individual rods during a second compounding step [6]. PS/PPE here is one phase with lower nanotube affinity than PA66.

The reference systems for selective localization are immiscible. Sumita and co-workers watched carbon black by transmission electron microscopy (TEM) and found it either filling one phase or sitting at the interface, with interfacial energy deciding the outcome when the two viscosities were close [1]. They coined the term double percolation for what follows: the filler must percolate within its host phase, and the host phase must itself be continuous. For nanotubes, PC/SAN is the clearest case of migration. Whether MWCNT were premixed into PC or SAN, they ended up exclusively in PC, and the blends had much lower resistivity than either single polymer composite at the same loading [7].

3. Physical Mechanisms and Analytical Approach

Above the threshold the direct current conductivity of a filled polymer follows a scaling law

sigma = sigma_0 (phi - phi_c)^t for phi > phi_c,

where sigma is the composite conductivity, sigma_0 is a prefactor with the dimensions of conductivity set by filler and contact resistances, phi is the filler volume fraction, phi_c is the critical fraction at which a connected cluster first spans the sample, and t is the conductivity exponent. Kirkpatrick's resistor network simulations gave a sharply defined concentration dependence of conductance near threshold, governed mainly by dimensionality [8]; for three dimensional lattices t lies between about 1.6 and 2.0. The power law holds only just above phi_c, where the network is a sparse backbone; well above threshold effective medium behaviour takes over and the curve flattens.

In nanotube composites the fitted exponents are often above 2, because charge passes by tunnelling across thin polymer layers between tubes rather than by metallic contact. Sheng's fluctuation induced tunnelling model treats a disordered conductor as conducting regions separated by thin insulating gaps, with thermal voltage fluctuations modulating each barrier [9]. Tunnelling resistance is exponential in gap width, so a spread of gaps broadens the network resistance distribution and inflates the apparent t. Bauhofer and Kovacs found that phi_c and t vary widely with nanotube type, polymer and dispersion method, and that a lower kinetic threshold, built by re-aggregation, is commonly reported [10].

Where the filler sits in a two phase blend is predicted at equilibrium by Sumita's wetting parameter [1],

omega_a = (gamma_filler-B - gamma_filler-A) / gamma_AB,

where gamma_filler-A and gamma_filler-B are the interfacial tensions between the filler and polymers A and B, and gamma_AB is the interfacial tension between the two polymers. For omega_a > 1 the filler is predicted in phase A, for omega_a < -1 in phase B, and between those bounds at the interface. The relation assumes a filler small compared with the blend domains, interfacial tensions estimated from surface energy components, and mixing long enough for equilibrium.

In practice the interfacial tensions are built from contact angle data, each surface energy split into dispersive and polar parts and combined by a geometric or harmonic mean rule. The nanotube value is taken from one or two literature sources and varies with purification and surface oxygen, and the two rules can give omega_a of opposite sign for the same inputs. Gültner and co-workers found that in PC/SAN with a reactive maleic anhydride copolymer, MWCNT moved from PC into the SAN rich phase although surface energies gave little indication and PC stayed the more viscous phase; they put it down to coupling between nanotube surface groups and the anhydride [16]. Chemistry beat thermodynamics and viscosity.

The equilibrium assumption is the weak one. The kinetic competitor is the transfer rate across the interface, set by the viscosity of each phase, the viscosity ratio, the mixing sequence and the shear history; a filler premixed into the more viscous phase may stay trapped there throughout compounding. Göldel and co-workers argued that slender particles are unstable at an interface and cross it quickly (the Slim-Fast mechanism), and tested this by watching MWCNT and carbon black transfer from SAN into PC [11].

For PS/PPE none of this applies unless two phases exist. In a single phase melt the variables reduce to local viscosity, which rises steeply with PPE content and slows nanotube diffusion and re-aggregation, mixing energy, which breaks agglomerates but shortens tubes, and time at rest above the glass transition, when tubes reconnect. What the mixer starts from is shown in Figure 2: as-produced nanotubes arrive as an entangled felt of bundles, held together by van der Waals forces, and every tube that ends up isolated in the melt had to be pulled out of a mat like this one.

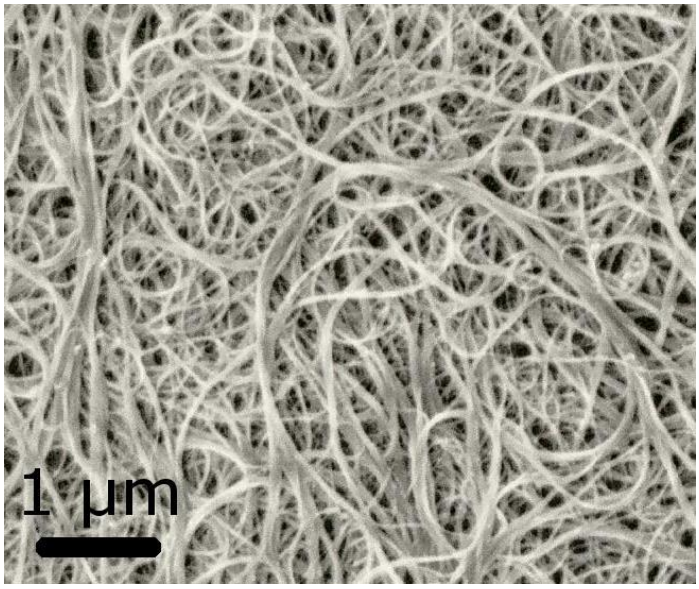


Figure 2. Scanning electron microscope image of carbon nanotube bundles, scale bar 1 micrometre. This is a bare nanotube mat, not a PS/PPE composite; it shows the entangled bundle state that melt mixing must break up before single tubes can disperse. Image: Materialscientist, Wikimedia Commons, CC BY-SA 3.0, https://commons.wikimedia.org/wiki/File:CNTSEM.JPG

The felt is the agglomerate that screw speed acts on, and the next section shows what that did in the one PPE/PS study.

4. Results from the Literature and Scientific Discussion

Table 1 collects the reported localization and threshold data. Only one row is PPE/PS with nanotubes. One more has PS/PPE inside a ternary blend, and the rest are immiscible PS based or related blends, there for mechanism rather than as substitutes.

Table 1. Reported nanotube localization and percolation behaviour in PPE/PS and reference blends

Blend and processing route	Composition and filler	Reported filler location	Reported threshold or conductivity	Ref.
PPE/PS miscible blend, twin screw extruder, masterbatch at 600 rpm diluted at 400 and 600 rpm	Miscible PPE/PS, 0 to 4 wt% MWCNT	Dispersed through single phase matrix; larger agglomerates at higher screw speed	phi_c = 0.4 wt%; about 12 orders of magnitude rise at 0.5 wt%	[5]
PA66/(PS/PPE) ternary blend, three compounding sequences	PA66 with miscible PS/PPE, CNT	Mainly in PA66; CNT premixed in PS/PPE transferred to PA66 as individual rods	Premixing in PS/PPE then adding PA66 gave highest conductivity; higher than PA66/CNT at same loading	[6]
PC/SAN co-continuous, microcompounder, premixed in PC or SAN or all together	PC/SAN, low MWCNT content	Exclusively in PC regardless of addition route	Much lower resistivity than PC or SAN composites at same loading	[7]
PP/PS, internal mixer, nanotubes added to preformed blend	PP:PS from 10:90 to 90:10 vol%, 1.0 vol% MWCNT	PS phase and interphase at all compositions (TEM)	Not reported; PS/CNT binding energy 30.6 to 32.6 kcal/mol versus 26.6 to 28.3 for PP	[12]
PP/PS co-continuous, melt mixed, then slow cooled or held at 135 °C	50/50 wt% PP/PS, 0 to 2 wt% MWCNT	PS phase	phi_c about 0.6 wt% (PP/MWCNT), 0.28 wt% (blend, fast cooled), 0.06 to 0.08 wt% after thermal treatment	[13]
PEO/PE co-continuous, MWCNT prelocalized in PE, mixing time varied	40:60 PEO/PE, 0.5 to 3 vol% MWCNT, with or without 6% PEMA	Migrated from PE to PEO with time (SEM)	22.1 S/m at 3 vol%, 1 min; 68.7 S/m compatibilized at 5 min	[14]

Blend and processing route	Composition and filler	Reported filler location	Reported threshold or conductivity	Ref.
PPE/HDPE droplet and co-continuous, MWCNT prelocalized in HDPE, 5 and 10 min	20:80 to 80:20 PPE/HDPE, 1 wt% MWCNT, 1 to 3 wt% SEBS	In HDPE without compatibilizer; migrated into PPE with SEBS (TEM)	Conductivity up 2 to 3 orders to 40% PPE, then down by up to 12 orders; SEBS raised 80:20 blend by about 4 orders	[15]

The table makes the asymmetry plain. In the immiscible systems the filler moves, and the move is seen. Göldel's PC/SAN result is the strongest case: the final location does not depend on the starting one, the signature of a thermodynamic preference reached within the mixing time [7]. A companion study showed MWCNT reaching PC faster than carbon black from the same SAN precompound [11]. In PP/PS the nanotubes end up in PS and at the interphase at every composition; simulated binding energies were about 4 kcal/mol higher for PS/CNT than for PP/CNT [12]. And Strugova and co-workers showed how far the threshold falls once nanotubes are confined to one continuous phase: from about 0.6 wt% in PP/MWCNT to 0.28 wt% in the co-continuous blend, and to 0.06 wt% when slow cooling let PP crystals push the tubes into a smaller volume [13].

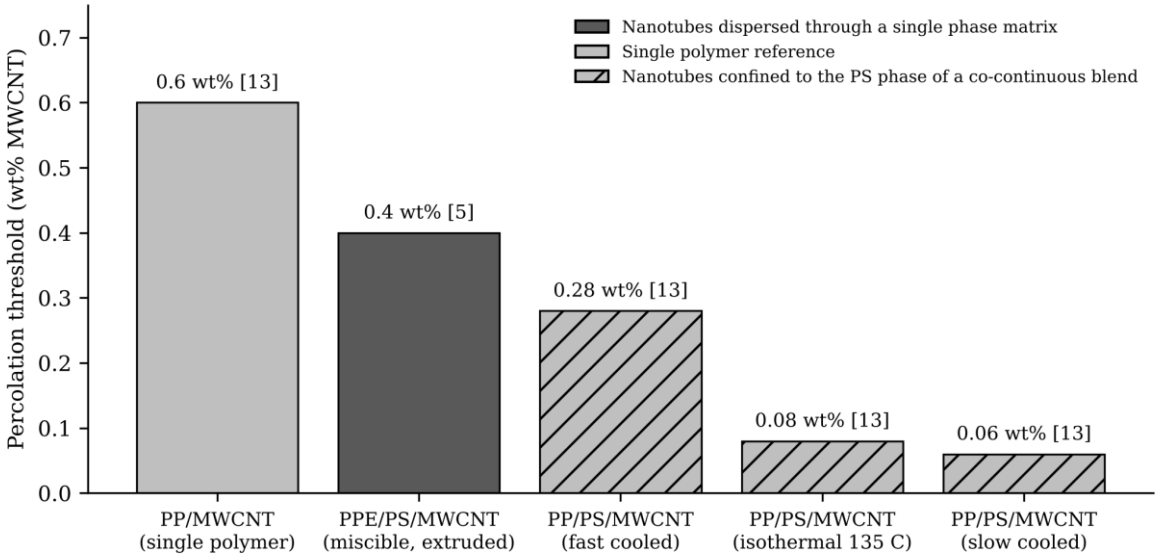


Figure 3. Reported percolation thresholds (wt% MWCNT) for the miscible PPE/PS composite of Sathyanarayana et al. [5] and for the PP/MWCNT and co-continuous PP/PS/MWCNT composites of Strugova et al. [13], the one source in Table 1 that gives thresholds for a single polymer and its blend on the same nanotubes. Dark bar: nanotubes dispersed through a single phase matrix. Hatched bars: nanotubes confined to the PS phase of a co-continuous blend, with and without thermal treatment.

In Figure 3 the miscible PPE/PS composite sits at 0.4 wt%, between the single polymer value and the untreated blend, and nowhere near the 0.06 to 0.08 wt% reached once PP crystallizes around the PS network. The comparison is not exact (different polymers, different mixers), but the gap survives that. Confinement to one phase does what dispersion in one phase cannot.

That 0.4 wt% [5] is typical of single polymer composites with well dispersed industrial MWCNT, inside the range Bauhofer and Kovacs tabulated [10]. It is what one expects of a single phase: the filler has nowhere to migrate, and the whole volume must be filled to the statistical threshold. The twelve order rise between 0.4 and 0.5 wt% is steep, pointing to a narrow spread of agglomerate sizes rather than a gradual kinetic network.

No t can be fitted to those points; [5] gives only the threshold and one conductivity step. But the pattern across nanotube composites is clear from Bauhofer and Kovacs [10]: fitted exponents scatter well above the lattice band of 1.6 to 2.0 from Kirkpatrick's networks [8]. In a tunnelling network each contact resistance depends exponentially on its gap, so the backbone carries a wide spread of link resistances; Sheng's model describes one such link [9], and many in series and parallel make the conductance near threshold fall faster than the cluster count predicts, read as a larger t. A high t in PS/PPE would say more about the polymer layer between tubes than about lattice geometry.

Put those numbers into the scaling law and the result is Figure 4. The matrix sits at a flat baseline until phi reaches phi_c, then the conductivity climbs through many decades within a few tenths of a percent and bends over.

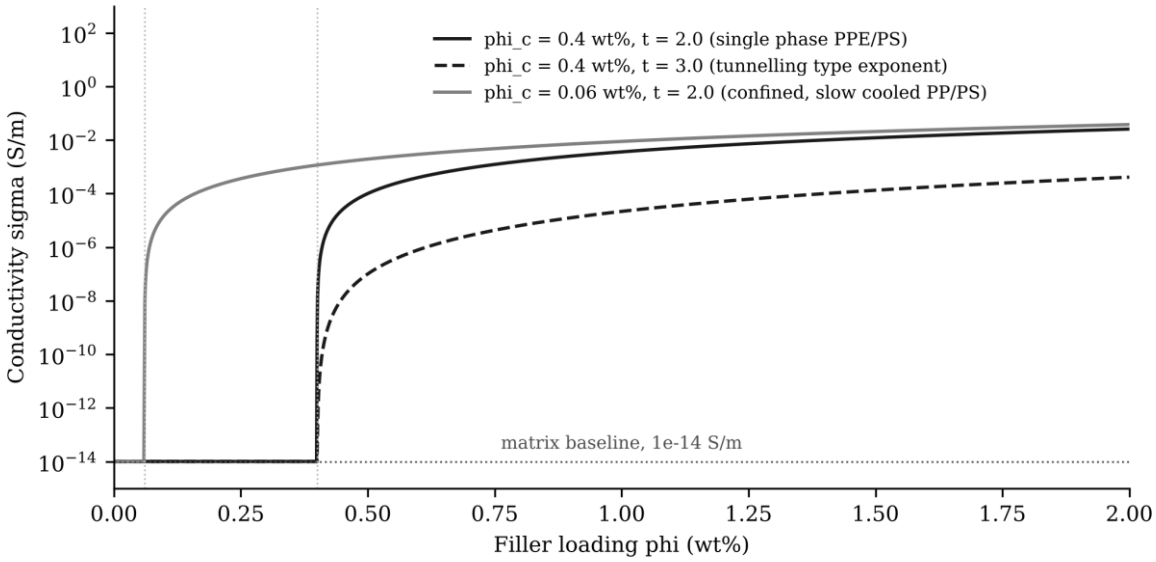


Figure 4. Model illustration of the scaling law sigma = sigma_0 (phi minus phi_c)^t, not measured data. Parameters: sigma_0 = 100 S/m (illustrative), phi in wt% with (phi minus phi_c) taken as a fraction, matrix baseline 1e-14 S/m (illustrative) below threshold. Solid black: phi_c = 0.4 wt% (the PPE/PS/MWCNT threshold of [5]) with t = 2.0. Dashed black: same phi_c with t = 3.0, the kind of exponent reported when tunnelling dominates [10]. Grey: phi_c = 0.06 wt% (the slow cooled co-continuous PP/PS/MWCNT threshold of [13]) with t = 2.0. Dotted verticals mark the two thresholds.

Two features deserve a look. Raising t from 2 to 3 at the same threshold pulls the whole curve down by two to four decades, so a tunnelling dominated composite can percolate at the same loading as a lattice-like one and still conduct far less; a threshold alone is a poor specification. And moving phi_c from 0.4 to 0.06 wt% shifts the steep rise to a loading seven times lower without changing the plateau. The comparison with the twelve order jump that [5] measured between 0.4 and 0.5 wt% is loose, since sigma_0 here is a guess.

Is the power law the right tool once length and alignment vary? Not on its own. Ayala-Ruano and Oliva-Aviles built a resistor network model in which nanotubes are generated stochastically with set length, degree of orientation and waviness, and each tube to tube link is assigned a resistance for direct contact, tunnelling or intrinsic conduction [18]. In their model tunnelling, not direct contact, was the main driver of conduction at the percolation stage, which fits the high exponents discussed above. And longer, straighter, moderately aligned tubes gave more conducting paths, so the threshold is not a property of the loading alone. A single fitted phi_c and t from a plot like the one in [5] describes that plot and nothing else; it carries no information about tube length after extrusion or about how flow has oriented the network, and both change with screw speed. A threshold measured on a compression moulded plaque may not transfer to an injection moulded part from the same pellets.

Screw speed was the one processing variable in that study that acted on the network, and it acted through dispersion: more large agglomerates at 600 rpm than at 400 rpm [5]. Higher shear in a viscous PPE rich melt breaks agglomerates but shortens tubes, and shorter tubes need more contacts to percolate.

The ternary PA66/(PS/PPE) results [6] show what migration looks like when PS/PPE is a phase, not the matrix. Nanotubes left the miscible blend for PA66 as individual rods, and premixing them in PS/PPE before adding PA66 gave the best dispersion and conductivity. The transfer step itself disperses the filler; tubes crossing an interface one at a time cannot drag an agglomerate along. PS/PPE gets this only when a third polymer supplies the interface.

Mixing time has an optimum when the filler starts in the wrong phase. In PEO/PE, conductivity peaked at 1 min and fell as nanotubes crossed into PEO; a compatibilizer moved the peak to 5 min [14]. In PPE/HDPE a block copolymer let nanotubes enter PPE and raised the 80:20 blend's conductivity by about four orders of magnitude [15]. Both put the direction down to affinity and the rate down to interface area, compatibilizer and viscosity. These mechanisms would operate in PS/PPE only if PS rich and PPE rich domains survived compounding. The two relaxation peaks in powder mixed blends [3] show such domains can exist, but no study reports nanotubes partitioning between them. Interphase migration in PS/PPE is neither demonstrated nor excluded. It is unobserved, and the driving force would be small, the two polymers being similar aromatic chains with near zero interfacial tension.

No verified source reports singlewalled nanotubes in PPE/PS. Their smaller diameter can lower the threshold, but stronger bundling makes dispersion in a viscous PPE rich melt harder. The numbers above are MWCNT data.

5. Practical Implications and Limitations

For a formulator the implications are direct. In plain PS/PPE the threshold is set by dispersion quality and tube length, not by phase partitioning. The variables that matter are screw speed, residence time, melt temperature (which with the PPE fraction sets viscosity) and the dilution route. The 0.4 wt% threshold for extruded PPE/PS [5] is a fair benchmark. A lower value should not be expected from selective localization alone.

Rheology offers a processing diagnostic. A nanotube network restrains the melt before it conducts, so the low frequency storage modulus turns solid-like at or below the electrical threshold. Du and co-workers saw this in SAN/PPE, where 2.5 wt% MWCNT in SAN gave solid-like low frequency behaviour, and used complex viscosity to follow where the tubes sat [17]. Lee and co-workers ranked dispersion across compounding sequences in PA66/(PS/PPE)/CNT by a nonlinear to linear viscoelastic

ratio; the sequence with the highest ratio was also the most conductive [6]. A test on the extrudate thus shows whether the network is forming before moulding.

Impedance spectroscopy adds a second diagnostic, and a recent study shows it can report where the filler sits as well as whether it percolates. Dadashi and co-workers built co-continuous PP/EVA blends (40/60 vol%) from separate PP and EVA masterbatches holding 1, 2 or 3 vol% carbon black, so that the split of filler between the two phases could be set at 1/3, 2/2 or 3/1 vol% while the total stayed fixed [19]. Electron and atomic force microscopy confirmed co-continuity throughout. The impedance spectra then told the phases apart. An uneven split, with 3 vol% in PP and 1 vol% in EVA, lowered the real part of the impedance and pushed the cut-off frequency upward, which the authors read as higher capacitance in the Nyquist plot; and inverting the blend ratio to 60/40 PP/EVA at a 2/2 split raised the capacitance 38 fold, which they attributed to the lower impedance of the carbon black filled PP phase once PP became the larger continuous network. Carbon black is not a nanotube, and PP/EVA is not PS/PPE. But the method transfers. For a PS/PPE composite with a third polymer, an impedance sweep on the compounded material could show whether nanotubes have crossed into the partner phase without waiting for TEM, on the same sample used for the dc measurement.

If a lower threshold is needed, the data favour a third, immiscible polymer. The PA66/(PS/PPE)/CNT results [6] and the PPE/HDPE results [15] show that a PS/PPE or PPE phase can host or release nanotubes depending on partner polymer and compatibilizer. The compounding sequence then becomes the control variable: premix in the low affinity phase and let transfer disperse the filler, or premix in the high affinity phase and stop before the tubes leave. Mixing time must match the migration kinetics, minutes in PEO/PE [14].

Several limitations apply. The only PPE/PS nanotube study used MWCNT and reported no viscosity ratio, interfacial tension or fitted t. Thresholds in Table 1 mix units (wt% and vol%) and geometry (droplet and co-continuous), so strict comparison is impossible. And the equilibrium prediction says nothing about the time to reach it; two samples with the same omega_a can show opposite localization after a short mix.

6. Conclusion

Nothing in the published record shows nanotubes choosing between a PS rich and a PPE rich region; the single compounded PPE/PS composite behaves like a filled single polymer, and the one route to a low threshold that the data support is a third, immiscible polymer, with the transfer step across its interface doing the dispersing. In the immiscible pairs where migration is seen, affinity picks the destination while viscosity, aspect ratio and mixing time pick the speed, and both rheology and impedance can follow the move without a microscope. What remains open is whether incompletely mixed PS/PPE, with its two mechanical relaxation peaks, ever holds domains long enough for nanotubes to partition between them. Nobody has looked. A compounding run stopped early, with TEM and an impedance sweep on the same extrudate, would settle it.

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