

## Benefits of Using OBSA in Various Applications

### Abstract

OBSA and its sodium salt, NaOBS, have untapped potential in a variety of applications that at least partly stems from being among the most (if not the most) hydrophilic surfactant amphiphiles in their structural class. In particular, this technical whitepaper highlights the value-added differentiation of OBSA/NaOBS in three mission-critical representative use categories, namely hydrophilization (e.g., wetting), dispersion stabilization, and solubilization.

### Background

4-Octylbenzenesulfonic acid<sup>i</sup> (OBSA; CAS# 17012-98-5) is a brown-hued liquid with melting and boiling points of approximately<sup>1</sup> 116 °C and 351 °C, respectively. Shown below in Figure 1, OBSA contains a sulfo group and linear eight-carbon (C<sub>8</sub>) alkyl chain attached to a benzene ring. In water-containing solutions, the OBSA and/or dissociated-form OBS<sup>-</sup> molecule C<sub>8</sub> tails' preferential orientation at the

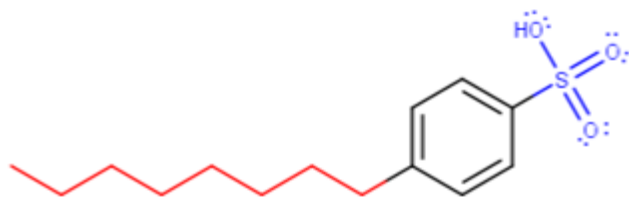


Figure 1. Structure of OBSA.

interfaces of and toward nonpolar regions (e.g., air, decane) complements—in the absence of, e.g., electrostatically attractive interactions or chemisorption—the sulfo/SO<sub>3</sub><sup>-</sup> headgroups' positioning toward the aqueous phase<sup>2,3</sup>. Such phase-bridging stemming from OBSA's amphiphilic<sup>ii</sup> character, which is further balanced by the benzene ring hydrophobicity, leads to surface and interfacial tension (IFT) reduction—a standalone application that can also be used as a lead-in to dispersion (i.e., suspension, emulsion) stabilization besides solubilization<sup>2,3</sup>. As such, although OBSA, in the form of a non-neutralized acid, affords the formulator maximum flexibility in achieving full conversion (i.e., via Le Châtelier's Principle<sup>4,5</sup>) to sulfonate molecules with the base(s) of their own choosing, Varsal can also offer and formulate salts (e.g., Na<sup>+</sup>OBS<sup>-</sup>; CAS# 6149-03-7) for direct purchase. Compared to Na-C<sub>6</sub>H<sub>13</sub>- and -C<sub>7</sub>H<sub>15</sub>-BS, NaOBS is widely considered, as a review of the patent literature would reveal, to be the compound for which applicable (to those inventions) surface activity, including micelle formation<sup>6</sup> at and above a CMC (10–14.7 mM)<sup>7,8</sup> and Krafft point  $T_k$  (18–26 °C)<sup>9,10</sup>, in its homologous series begins. Accordingly, it is the most hydrophilic surface-active agent (i.e., surfactant) in its alkylbenzenesulfonate class<sup>2,11</sup>.

With longer-tailed homologues (e.g., NaDDBS) often<sup>11,12</sup> containing structural impurities, if not comprising blends, one advantage of OBSA and derived salts is their isomeric<sup>13</sup> and homologous<sup>14</sup> purity. This makes them useful for research studies seeking to avoid mixed adsorption behavior that could otherwise, for instance, complicate interfacial area measurements<sup>13</sup>. Similarly, with its shorter tail pointing to, besides a lower  $T_k$ , greater water solubility (4.9 g/L at 20 °C)<sup>15</sup> and a quicker diffusion to—along with greater adsorption or even equilibration<sup>16</sup> at—interfaces for lower tensions at shorter times (dynamic wetting), NaOBS could be more helpful than competitors in room-temperature applications involving fast surface creation rates (e.g., high-speed lithographic printing<sup>17</sup>)<sup>2</sup>. Compared to NaDDBS, NaOBS has been, as seen<sup>18</sup> with activated carbon at pH  $\ll$  pH<sub>PZC</sub>, a more effective hydrophobizing agent and flocculant for +-charged substrates due to their strong interactions with, and polarization of, the benzene ring  $\pi$ -electrons giving a more-parallel surfactant adsorption—and hence greater cross-sectional area for the, and surface saturation occurring with a smaller number of, longer-tailed surfactant molecules<sup>2</sup>. Meanwhile, at adsorptive concentrations sufficient for charge-reversal and increased negative, e.g., zeta potential, at least

<sup>i</sup> Another common name is *p*-octylbenzenesulfonic acid.

<sup>ii</sup> Another commonly used descriptor is "amphipathic".

one of the mechanisms for OBSA's noticeably greater removal than DDBSA of foreign contaminants from photoresist pattern surfaces (OBSA also gave identical line-width roughness) after spin-drying when paired with the same alkyleneoxy-based nonionic surfactant in post-development rinsing could have been electrostatic repulsion between particles and substrate<sup>2,19</sup>. This could similarly have been one of NaOBS's roles in different cleaning solutions, also involving void cleaning, for LCD panels<sup>20</sup>, while NaOBS-induced repulsion can separate more hematite from glass than can dodecylpyridinium chloride<sup>21</sup>. For an example of a non-aqueous application, NaOBS in methanol solvent is noted<sup>22</sup> to be a unique combination for providing high metal oxide (e.g., Al<sub>2</sub>O<sub>3</sub>) particle dispersibility and aggregation suppression (besides exemplary hydrophobizing action). When this dispersion is poured on top of water, a surface monolayer (later transferred to, and coated on, dry etching substrates and used as an etching mask) of particles that are two-dimensionally close-packed with high accuracy could then be easily obtained<sup>22</sup>.

OBSA/NaOBS utility in hydrophilization, dispersion stabilization, and solubilization is considered in more detail below.

## Representative Cases of OBSA as a Value-added Differentiator

### Usage 1: Hydrophilizing Agent

As a hydrophilizing (e.g., wetting, spreading) agent, NaOBS at  $\sim 1.3 \times 10^{-3}$  M has equilibrated "extremely rapidly" (i.e., effectively instantaneously) to yield water droplet surface tensions of  $\sim 46$  dyn/cm (mN/m)<sup>23</sup>. Similarly quick equilibrations from diffusion-controlled adsorption could occur at other aqueous-nonpolar interfaces, with lower IFTs between water and saturated hydrocarbon (e.g., hexane) phases possible from increased adsorption efficiency and, maybe slightly increasing with alkane length, effectiveness<sup>2,24</sup>. With the comparatively poor[er] resistance of longer homologues (e.g., Na-C<sub>10</sub>H<sub>21</sub>-BS) to counterion-induced precipitation from water, NaOBS could be especially useful for optimal wetting in, besides hard water, highly inert-salt-concentrated—e.g., NaCl, Na<sub>2</sub>SO<sub>4</sub>—and/or strongly alkaline (e.g., pH  $\geq 10$ ) applications<sup>2,25</sup>. Examples of the latter might include oven and drain cleaners, scouring and kier boiling for textiles, metal cleaning, etc.<sup>25</sup>. At 0.1 g ( $\sim 0.37$  mM)<sup>26</sup>, OBSA or one of its salts was also used as a leveling agent to improve the planarization properties of water-soluble polymer coatings, with portions of the underlying annealed TiO<sub>2</sub>-layered substrates possibly<sup>27</sup> becoming more hydrophilic upon subsequent photomask-facilitated UV exposure. As a hydrophilizing etching-rate suppressant—and leveling agent through interfacial adsorption for, e.g., inhibition of H<sub>2</sub>-bubble-based surface roughening<sup>28</sup>—0.4 mM ( $\sim 0.01$  wt.%) OBSA<sup>iii</sup> in 5 wt.% aqueous NaOH<sup>iv</sup> at  $\sim 85$  °C allowed for, on account of OBSA's lack of structural impurities, precise and reproducible (with little variation between lots) pattern formation in anisotropic wet etching<sup>14,29,30</sup>. NaOBS has also found application and success in ink-jet textile printing<sup>31</sup>.

Without tension-reducing additives (e.g., electrolyte, co-surfactant), NaOBS can yield  $\gamma_{\text{CMC}} = 40.9$  dyn/cm in aqueous solution (30 °C)<sup>32</sup>. A reduction to  $\gamma_{\text{LV}} \sim 36$  dyn/cm at 0.15 M<sup>6</sup> mirrored<sup>2</sup> by an IFT reduction could be helpful in SWNT dispersion stabilization, as seen in the following usage section, and solubilization. Though featuring a higher  $T_k$ , a K<sup>+</sup> salt could better reduce surface tension and IFT due to the more-tightly-bound potassium counterion's greater neutralization of surfactant ion (OBS<sup>-</sup>) charge<sup>2</sup>. A surfactant blend of mole fractions 25% for sodium lauryl sulfate (SLS) and 75% for NaOBS gives  $\gamma_{\text{CMC}} = 31.5$  dyn/cm (30 °C)<sup>32</sup>. Such a system could be advantageous in hard surface (i.e., nonporous, nongranular solids or films) wetting given that, since these surface areas are relatively small[er], equilibrium-like conditions are usually obtained<sup>2</sup>. However, given SLS's and NaDDBS's considerable wetting agent use (e.g., for granule breakdown) in patented agrochemical formulations, the NaOBS/SLS mixed wetter could also be evaluated for solid formulations (e.g., WP, WG, SC) containing a non-wetting dispersant such as sodium naphthalenesulfonate formaldehyde condensate or lignin sulfonate<sup>2,33</sup>. OBSA (with, e.g., Na<sub>2</sub>HPO<sub>4</sub>)

<sup>iii</sup> In the presence of silicon oxoacid (silicate) salts, even-lower amounts might be optimal. Without silicate salts, 0.1 wt.% could be too much.

<sup>iv</sup> Per the invention, KOH could also be used.

was otherwise repeatedly employed in capsule suspension (CS) formulations, which when emulsified with oil dispersions of neonicotinoids resulted in innovative “OD+CS” pesticide formulations<sup>34</sup>.

For one particularly innovative application, the improvement highlight of a recently (ca. 2020) patented<sup>35</sup> production method for allergy-resistant low-protein vulcanized foamed latex is a 1.2 : 1 w/w blend of NaOBS and sodium hexadecylbenzenesulfonate—which, with their solubility cooperativeness giving<sup>2</sup> closer-packed and hence mechanically stronger (e.g., against coalescence) interfacial adsorption films, could separately make for an effective emulsifying agent. Besides NaOBS’s potential for solubilizing its more-oil-soluble partner in the polar phase, the inventors found this pairing playing a synergistic effect with the cetylpyridinium bromide, polyether-modified organosiloxane (nonionic surfactant), and foam stabilizer<sup>v</sup> (e.g., potassium oleate)—giving “obviously improved” foaming from lower surface tension courtesy of a boosted hydrophilizing action<sup>2,35</sup>. With regard to foam stabilization, the even-stronger intermolecular cohesive forces (i.e., enhanced interfacial films) gave rubber products with stable and uniform foam pores, in addition to markedly improved mechanical properties and fewer internal defects<sup>2,35</sup>. The small cohesive strength difference between the molecular chains of NaOBS, CPB, and siloxane may have been what led to them being mutually well-diffused<sup>vi</sup>, which in turn was a source of: the aforementioned tighter intermolecular interaction forces between the five surfactants, and preventing the auxiliary zinc oxide foaming contribution from being unstable due to particle agglomeration<sup>23,35</sup>.

For low-pH cleaning formulations (e.g., dairy cleaners), 1 wt.% OBSA could be useful—yielding  $\gamma_{LV} \sim 28$  dyn/cm<sup>vii</sup>, which was lower than for NBSA and DBSA (same wt.%, setup) in the same molecular simulations study<sup>36</sup>. One possible source for the additive-less tension achieved is dimer formation from H-bonding between OBS<sup>−</sup> and OBSA molecules, perhaps with vesicle formation, as pH decreases with increasing OBSA addition<sup>37</sup>. OBSA’s tension reduction combined with aggregate assembly could be applied in certain non-aqueous solvents (e.g., tetraglycol, 1,2-propanediol) for dissolution-based photoresist stripping without etching or pitting of exposed metal layers, given 4-ethylbenzenesulfonic acid’s demonstrated usefulness there<sup>36</sup>. The aggregate-based hindrance of headgroup interaction (these groups being pointed inward, toward their aggregate interiors) with relatively polar metal surfaces was thought to inhibit corrosion<sup>36</sup>.

### Usage 2: Dispersion Stabilizer

After interfacial adsorption and a possible application of shear, OBSA salts could also be used for electrostatic-repulsion-based suspension and emulsion stabilization from electrical double-layer overlap between dispersed particles<sup>2,3,38</sup>. In this case, keeping the [inert electrolyte] below 0.1 mol/L has been a useful rule of thumb for avoiding, e.g., flocculation leading to aggregation/coalescence from too much charge-shielding-based reduction of magnitude of potential<sup>3</sup>. Compared to SLS’s aqueous suspension of nanotubes (0.5 mg/mL) at the optimum nanotube-surfactant ratio<sup>viii</sup> containing tube bundle sediment after 5 days, NaOBS fully stabilized more SWNTs (8 mg/mL) in the same research study<sup>39</sup> against both sedimentation and aggregation for at least 3 months even up to 10–20 times its CMC<sup>ix</sup>. Given the substantial vdW attractions of bare SWNTs, NaOBS could hence be advantageous for dispersing other small-diameter—i.e., energetically favoring alkyl chain adsorption parallel to the cylindrical axis, followed by potential hemimicelle<sup>40,41</sup> formation at higher [NaOBS]—rod-like hydrophobic particles, perhaps even non-tubular structures given that, besides graphite, the  $\pi$ -like stacking<sup>39</sup> of molecule benzene rings has also been observed on coal<sup>42</sup>. In emulsion polymerization, polychloromethylstyrene nanoparticles of size  $12.0 \pm 2.3$  nm have been prepared at high conversion rate (ca. 99%) utilizing NaOBS without co-surfactants<sup>43</sup>. By better protecting against coalescence, increasing [NaOBS] led to decreasing particle diameter and size

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<sup>v</sup> It was not particularly limited, so different surfactants (e.g., castor oil soap, polyethylene glycol) could be selected to achieve similar effects.

<sup>vi</sup> For reference, cetylpyridinium chloride under diffusion-controlled adsorption, like NaOBS, has been noted to rapidly equilibrate at interfaces.

<sup>vii</sup> The measured surface tension for water was 75.64 dyn/cm.

<sup>viii</sup> Somewhere in-between 1:5 and 1:10 w/w for each surfactant examined, and determined by mixing 0.01 mg/mL nanotube dispersions with various surfactant concentrations and seeing when the tubes remained dispersed after sonication.

<sup>ix</sup> Given the 1:5–1:10 w/w ratio, stabilizing 8 mg/mL of SWNTs entailed 0.137–0.274 mol/L of NaOBS.

distribution, both of which also continued decreasing above the CMC<sup>43</sup>. Besides more safely allowing for high-shear applications, adding a non-ionic co-surfactant for steric (and thus, when combined, *electrosteric*) stabilization would better protect against pH-induced destabilization and potentially allow, for hydrophilizing, greater electrolyte additions<sup>3,44,45</sup>.

### Usage 3: Solubilizing Agent

At and above their CMCs, OBSA salts could also be used for aggregate-based (e.g., micelles) solubilization of substances in the presence of aqueous solution<sup>2,3</sup>. One could increase temperature, and forego an inert salt addition, to increase the extent of solubilization for polar materials<sup>2,46</sup>. Mixed NaOBS-PEG anionic-nonionic aggregates have recently (ca. 2020) proven quite effective in distilled-water detergents with ethanol auxiliary for contaminant (mustard, ink, coffee, wine) removal from corrugated, hard surfaces of laminated films<sup>47</sup>. Also, NaOBS's complexes with polyethylene glycol have "high solubilizing power"<sup>48</sup> for Yellow OB that increase with PEG's degree of polymerization<sup>2</sup>. Solubilization of nonpolar materials, including other oil-soluble azo dyes (e.g., Sudan Red G), should become particularly rapid with temperature approaching a nonionic cloud point<sup>2</sup>. A detailed study (ca. 2014) on NaOBS's solubilizing properties with regard to, and this surfactant's effects on the transport of a model hydrophobic drug (griseofulvin) from, swelling hydrophobic tablets may be found in the research literature<sup>49</sup>. Controlled release of active ingredients via capsule solubilization could<sup>50</sup> also be an advantageous application for middle-phase microemulsions of NaOBS-pentanol-dodecane-water NaCl, the Winsor III (i.e.,  $R = 1$ ) systems of which featured<sup>51,52,53,54</sup> ultralow IFTs (e.g.,  $10^{-3}$ – $10^{-2}$  dyn/cm) under [NaOBS + 1-pentanol] independent variable scans. A system (21 °C) of 1-pentanol : NaOBS = 2 (w/w), H<sub>2</sub>O : dodecane = 2, and NaOBS + 1-pentanol = 7 wt.% has an optimal salinity (NaCl) around<sup>54</sup> 7–8 wt.%, from which increasing [NaOBS + 1-pentanol] should<sup>2,3</sup> lead to a Winsor IV microemulsion that could be evaluated for soiled textile surface cleaning—especially with water-insoluble solid particles (e.g., polyalkenes) reducing the amount of surfactant required<sup>55</sup>. For ternary NaOBS-pentanol-water systems at 25 °C, one-phase ( $L_1$ ,  $L_2$ ) domain and lamellar liquid crystal (LLC) transitions have been characterized<sup>56</sup>.

In a patented (ca. 2020) zeolite production method applicable to many different crystal structure catalysts (including small-pore types such as CHA, SOD, PHI, etc.), NaOBS's LLC "nanoreactors" were utilized as templates for controlled nanoscale-shaping<sup>57</sup>, and successfully prevented any aggregation, of zeolite intermediates with quite large ( $\geq 200$ ) aspect ratios<sup>2,58</sup>. By solubilizing<sup>59,60</sup> water in the LLC-swelling (e.g., decane) solvent, NaOBS provided, inside its amphiphilic bilayers, hydrophilic reaction field "hosts" for 'bottom-up' synthesis (e.g., gram-scale yields) of the intermediates from the hosted precursors (e.g., TEOS leading to SiO<sub>2</sub> particles)<sup>2,3,58</sup>. Though 1-pentanol's lowering of interfacial film rigidity helped stabilize the LLCs of stacked lamellae through the accompanying<sup>61</sup> interlamellar Helfrich undulation repulsions—even hyper-swollen LLCs were stabilized, thereby allowing for, e.g., 1–2 nm nanosheet-like particles to still be obtained in high numbers (i.e., synthesizable amounts)—1-hexanol might allow for even higher aspect ratios<sup>62</sup> from enlarged mesopore sizes<sup>63</sup> to be achieved<sup>58</sup>. Meanwhile, compared to other amphiphiles (especially the alkylbenzenesulfonates), the presentation temperature(s) of the LLCs formed from NaOBS were found to be more suitable for reaction temperatures around 60–70 °C (possibly 40–80 °C as well)—such that NaOBS was found to optimize the zeolite intermediate synthesis step (and so dry-gel conversion and crystallization were performed separately, with a different structure-directing agent)<sup>2</sup> and was utilized in every illustrative example<sup>58</sup>. Given that polymerizations are often-enough carried out at 60–70 °C (e.g., AIBN initiator in thermal polymerization), NaOBS-pentanol self-assembly systems could thus be highly advantageous for directing polymerization selectivity in the bilayers besides template monomer polymerization in the interlamellar regions<sup>2,64</sup>. One could then corroborate the NaOBS-pentanol interfacial film stability, given polymerization's general potential for causing phase transitions<sup>64</sup>. As the lamellar mesophase behavior of aqueous NaOBS-pentanol systems with and without hydrocarbon solvent is well-characterized in literature<sup>65,66,67,68,69,70</sup>, one could also more readily modify known mixtures to produce desired product templates. In fact, compared to non-mesophase polymerizations, lamellar-phase

polymerizations could achieve potentially faster kinetics and more optimal dispersities ( $\bar{M}_w/\bar{M}_n$ )—particularly with polar monomers (whose lamellar-phase polymerizations, compared to other lyotropic mesophases of non-reacting surfactants, have been the fastest)<sup>64,71,72</sup>.

## Varsal Advantage

Varsal is a leading producer of extremely-high-purity OBSA, capable of producing both free acid and derived salt forms. We are differentiated from the competition as Varsal's proprietary manufacturing logistics processes allows us to produce consistent, stable, extremely-high-purity material—leading to maximal yield and product quality for Varsal's customers.

Low residual cationic impurities would be important for a number of reasons, particularly when a chelator or sequestering agent could unnecessarily complicate a desired application<sup>2,3,73</sup>. For the case of divalent cationic impurities (e.g.,  $\text{Ca}^{2+}$ ) in OBSA and derived salts, these reasons could more particularly, especially compared to monovalent impurities (e.g.,  $\text{Na}^+$ ), include avoiding reduced hydrophobizing action on  $+$ -charged substrates; inhibited electrostatic-based removal of particles from collector materials; and, reduced dispersion stability<sup>2,3</sup>. Even small amounts of  $\text{Ca}^{2+}$  can abruptly elevate  $T_k$ —which could be particularly detrimental to NaOBS's potential in room-temperature applications—and thereby inhibit solubilizing action and/or micelle formation<sup>2,10</sup>. The amphiphile would also be more susceptible to precipitation, perhaps particularly in applications already involving inert and/or pH-adjusting electrolyte (e.g., for hydrophilizing power increase)<sup>2,3,32</sup>. Regarding specific industrial applications, the presence of metallic impurities, whether monovalent or divalent, can have an adverse effect on the anisotropic etching of silicon<sup>74</sup>. The minimization of monovalent cationic impurity (e.g.,  $\text{Na}^+$ ) concentration would also be important so that counterion-related stabilization could be properly accounted for through user-added electrolyte (e.g., NaOH for *in situ* neutralization of OBSA)<sup>3</sup>. This would reduce the potential for stabilization errors stemming from, e.g., trying to add the “remaining” counterion but obtaining a skewed pH, which could adversely affect process yields<sup>3</sup>. Though on its own perhaps not as worrisome as divalent impurities, extra  $[\text{Na}^+]$  beyond the user-applied electrolyte could still lead to reagglomeration of dispersed particles if the total  $[\text{Na}^+]$  crossed over the critical threshold<sup>3</sup>.

Varsal is able to serve a wide variety of end-markets and applications, as our intimate knowledge of the manufacturing process allows us to custom-manufacture various grades of OBSA and derived salts tailored to our customers' requirements. Please contact us at [info@varsal.com](mailto:info@varsal.com) to learn more about how Varsal can help you solve your complex chemical and specialty intermediates challenges!

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