Aqueous Biphase Extraction for Processing of Fine Coal

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ABSTRACT

Ever-stringent environmental constraints dictate that future coal cleaning technologies be compatible with micron-size particles. This research program seeks to develop an advanced coal cleaning technology uniquely suited to micron-size particles, i.e., aqueous biphasic extraction. The partitioning behaviors of silica in the polyethylene glycol (PEG)/dextran (Dex) and dextran/Triton X-100 (TX100) systems have been investigated, and the effects of sodium dodecylsulfate (SDS) and dodecyltrimethylammonium bromide (DTAB) on solid partition have been studied. In both biphasic systems, silica particles stayed in the top PEG-rich phase at low pH. With increase in pH, the particles moved from the top phase to the interface, then to the bottom phase. At very high pH, the solids preferred the top phase again. These trends are attributable to variations in the polymer/solid and nonionic surfactant/solid interactions. Addition of ionic surfactants into these two systems introduces a weakly charged environment, since ionic surfactants concentrate into one phase, either the top phase or the bottom phase. Therefore, coulombic forces also play a key role in the partition of silica particles because electrostatic attractive or repulsive forces are produced between the solid surface and the ionic-surfactant-concentrated phase. For the PEG/dextran system in the presence of SDS, SiO₂ preferred the bottom dextran-rich phase above its pH PZC. However, addition of DTAB moved the oxide particles from the top phase to the interface, and then to the bottom phase, with increase in pH. These different behaviors are attributable to the fact that SDS and DTAB concentrated into the opposite phase of the PEG/dextran system. On the other hand, in the dextran/Triton X-100 system, both ionic surfactants concentrated in the top surfactant-rich phase and formed mixed micelles with TX100. Therefore, addition of the anionic surfactant, SDS, moved the silica particles from top phase to the interface or bottom phase. On the other hand, DTAB, a cationic surfactant, attracted the particles to the top phase in the pH range form 4 to 11, where in the DTAB-free system the solids either stayed at the interface or in the bottom phase.
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EXECUTIVE SUMMARY

The objective of this research project is to develop an aqueous biphase extraction process for the treatment of fine coals. Aqueous biphase extraction is an advanced separation technology that relies on the ability of an aqueous system consisting of a water-soluble organic polymer and an inorganic salt to separate into two immiscible aqueous phases. Differences in the hydrophobic/hydrophilic properties of particulates can then be exploited to effect selective transfers to either the top polymer-rich phase, or the bottom salt-rich phase. The goal of this experimental program is to identify process conditions that optimize the selective transfer of coal into the top phase while retaining the mineral matter in the bottom phase. An additional goal is to develop an improved coal-pyrite separation technique based on aqueous biphase extraction.

The partitioning behaviors of silica in the polyethylene glycol (PEG)/dextran (Dex) and dextran/Triton X-100 (TX100) systems have been investigated, and the effects of sodium dodecylsulfate (SDS) and dodecyltrimethylammonium bromide (DTAB) on solid partition have been studied. In both systems, silica particles stayed in the top PEG-rich phase at low pH. With increase in pH, the particles moved from the top phase to the interface, then to the bottom phase. At very high pH, the solids preferred the top phase again. These trends are attributable to variations in the polymer/solid and nonionic surfactant/solid interactions. Addition of ionic surfactants into these two systems introduces a weakly charged environment, since ionic surfactants concentrate into one phase, either the top phase or the bottom phase. Therefore, coulombic forces also play a key role in the partition of silica particles because electrostatic attractive or repulsive forces are produced between the solid surface and the ionic-surfactant-concentrated phase. For the PEG/dextran system in the presence of SDS, SiO2 preferred the bottom dextran-rich phase above its pH_PZC. However, addition of DTAB moved the oxide particles from the top phase to the interface, and then to the bottom phase, with increase in pH. These different behaviors are attributable to the fact that SDS and DTAB concentrated respectively into the opposite phase of the PEG/dextran system. On the other hand, in the dextran/Triton X-100 system, both ionic surfactants concentrated in the top surfactant-rich phase and formed mixed micelles with TX100. Therefore, addition of the anionic surfactant, SDS, moved the silica particles from the top phase to the interface or bottom phase. On the other hand, DTAB, a cationic surfactant, attracted the particles to the top phase in the pH range form 4 to 11, where in the DTAB-free system the solids either stayed at the interface or in the bottom phase.

INTRODUCTION

Coal represents the largest source of fossil energy in the U.S. This resource also represents the most readily accessible and socially acceptable substitute for oil and natural gas. In response to the stipulations of the Clean Air Act (1), advanced coal cleaning technologies are needed in order to more efficiently eliminate unacceptable levels of ash and sulfur from the run-of-mine coal prior to combustion. The ability to achieve mineral matter liberation from coal is fundamental to all separation techniques
that seek to produce super-clean coal. It has been concluded from the microscopic examination of a wide variety of U.S. coals that, in many cases, grinding to 10 \(\mu\)m or below may be necessary in order to achieve the necessary liberation (2). It is clear, therefore, that future advanced coal cleaning technologies must be capable of accommodating micron size particles.

This project seeks to investigate the feasibility of separating pyrite and mineral materials from fine coal by using aqueous biphase extraction. Since Albertsson’s pioneering work in the mid-1950s (3), aqueous biphase systems have gained a place among the important separation media in biotechnology (3-7). These two-phase systems are usually composed of a water-soluble polymer and another component, e.g., another water-soluble polymer, a surfactant, or an inorganic salt. Recently the application of aqueous biphase extraction has broadened to include the separation of ultrafine particle (8-14) and metal ions (15-17).

The partitioning behavior of many inorganic particles in aqueous biphase systems has been investigated in our laboratory (18-20). As in conventional liquid-liquid extraction (21-23), the partition of particles in aqueous biphase systems is dependent upon the physicochemical interaction between the solid surface and the liquid solution, rather than the bulk properties of the solid or solution such as density. Therefore, the selective partition of particles in different systems can be realized by controlling the surface chemistry of the solids, or by carefully choosing aqueous biphase systems with specific interaction between liquid phase components and the solids.

In previous paper, the partitioning behavior of silica in the PEG/\(\text{Na}_2\text{SO}_4\) system (19) has been studied. In this system, the particles prefer the top PEG-rich phase under all pH values, which is attributable to the specific polymer-solid interaction through hydrogen bonding between the surface silanol groups and oxygen atoms in the polymer chains. No information has been found in the literature about the partition of silica in the polymer/polymer or polymer surfactant aqueous biphase systems (8-14, 18-20). For comparison with our previous studies (19), this work will investigate the partition of silica in the Dex/TX100 and PEG/Dex system. In the polymer/nonionic surfactant systems, additions of ionic surfactant successfully modify the distribution of proteins (24-26). In this system, a weak charged environment can be introduced by adding ionic surfactants, which become concentrated in the top surfactant-rich phase and form mixed micelles with the phase-composing nonionic surfactant. Therefore, it has been demonstrated that a coulombic attraction or repulsion develops between the surfactant-rich phase and charged proteins. Addition of anionic surfactants moved negatively charged proteins into the bottom dextran-rich phase, and transferred positively charged ones into the top phase. The proteins showed opposite behavior in the presence of cationic surfactants (24-26). Accordingly, this report will also investigate the role of electrostatic interaction in the partition of silica in the polymer/polymer aqueous biphase system by adding ionic surfactants into the system.
RESULTS AND DISCUSSION

Effect of pH on Silica Partition in the PEG/Dextran System

Figure 1 shows the partitioning behavior of silica in the PEG/Dex system. From pH 2 to 7, silica particles preferred the top PEG-rich phase while there was increasing solids concentration at the interface with increase in pH. At pH 8, the particles mainly stayed at the interface, with some in the top phase. In the pH range from 9 to 11, the solids transferred predominantly to the bottom dextran-rich phase, the amount of silicon dioxide at the interface decreased with increase in pH. At pH 12, the solid moved to the top phase again. For purposes of mass balance, the particles at the interface were combined with the silica in the bottom phase.

It is known that PEG adsorbs on silica surface through hydrogen bonding between the H atom of the Si-OH group and the O atom in the PEG chain, and it is a typical polymer adsorption with high affinity (27-34). The neutral Si-OH sites favor the adsorption of PEG (29, 31-33). When Si-OH deprotonates to form Si-O⁻ above the pH_pzc of silica, the formation of hydrogen bonds on these sites is not favored because of the electrostatic repulsion between these sites and the partially negatively charged oxygen atoms in the PEG chain. It is reported that the adsorbed PEG layer on the surface partially desorbed into the solution at high pH values. This desorption process was attributed to the electrostatic repulsion between the polymer chain and the solid surface (35-37).

Dextran can also adsorb on silica, a result of hydrogen bonding between the surface silanol groups and glycosidic or hydroxylic acceptor sites in the repeating glucose units of the polysaccharide (38-41). Baudin et al (38) found that the adsorption isotherms showed an unusual shape. The adsorbed polymer could be easily desorbed through dilution. They attributed all these phenomena to the poor affinity of dextran for the silica surface. The results from Jucker et al (40) also indicated that the adsorption density of dextran on silica is very low.

According to the results available in the literature, in aqueous solution both PEG (27-37) and dextran (38-41) can adsorb on silica surface through hydrogen bonding. Therefore, when silica particles are dispersed in the PEG/dextran system, a competitive adsorption process occurs at the solid/liquid interface between these two polymers. Hence, the partition behavior of silicon dioxide is determined by the relative strength of the PEG/SiO₂ and Dex/SiO₂ interactions. From the above discussion, it appears that PEG interacts more strongly with silica surface than dextran, because the adsorption of PEG on surface shows high affinity (27-34) while the polysaccharide only shows an adsorption process with poor affinity (38-41). This is the reason why silica prefers the PEG-rich phase at low pH values. With increase of pH, more and more surface silanol groups begin to deprotonate and the surface acquires negative charges. Then, the hydrogen bonding between PEG and SiO₂ is limited because of the electrostatic repulsion between the negatively charged solid surface and the partial negative charges on the oxygen atoms in the polymer chains. This repulsion force was demonstrated by the observed decrease in PEG adsorption on SiO₂ with increase in pH (35-37). Thus, the PEG/SiO₂ interaction
becomes weak, even possibly less than that for Dex/SiO$_2$. Accordingly, as pH increases, the particles begin to transfer from the PEG-rich phase to the interface (at pH 8), then to the dextran-rich phase (at pH 10 and 11).

The result at pH 12 is very interesting; the particles go into the top phase again. This behavior is difficult to explain from current literature about adsorption of PEG and dextran on silica. From this experimental result, it can be deduced that the interaction between dextran and silica is less than that between PEG and silica. Since the adsorption of PEG on silica decreases with increase of pH (35-37), it appears that the adsorption of dextran on silica is suppressed by a high pH environment. However, this needs to be demonstrated with adsorption experiments at high pH.

**Effects of Ionic Surfactants on Silica Partition in the PEG/Dextran System**

*SDS.* Figure 2 shows the partition of silica in the PEG/dextran system in the presence of 1.0 g/L SDS. In the pH range from 3 to 12, the particles mainly stayed in the bottom dextran-rich phase. At pH 2, there were solids in both the top and bottom phases.

In the PEG/dextran system, SDS mainly partitions into the top PEG-rich phase, which is due to the formation of polymer/surfactant complexes (42). Since silica is negatively charged above its pHPZC (about pH 2) (43), there is electrostatic repulsion between the SDS in the top phase and the solid particles. Then, it is expected that this repulsive force will suppress the adsorption of PEG on silica surface because SDS mainly concentrates in the PEG-rich phase. The work of Maltesh and Somasundaran shows that PEG adsorption on silica surface is decreased by addition of SDS (44). Therefore, the partition of oxides in the top phase is not favored in the presence of SDS. The results in Figure 2 show that from pH 3 to 12, silica particles mainly stayed in the bottom dextran-rich phase. At pH 2, silica surface is almost neutrally charged because of the coincidence with the pHPZC. Therefore, the repulsion between SDS and silica surface is reduced, and the particles dispersed in both the top and bottom phases.

*DTAB.* Figure 2 also shows the partition of silica in the PEG/dextran system in the presence of 0.1 g/L DTAB. From pH 2 to 6, the particles mainly stayed in the top PEG-rich phase, with some at the interface. In the pH range of 7 to 10, the oxides transferred to the bottom dextran-rich phase, the amount of solids at the interface decreased with increase in pH. At pH 11 and 12, almost all the silicon dioxide particles moved to the dextran-rich phase.

In the PEG/Dex system, in contrast to SDS, DTAB prefers the bottom dextran-rich phase (42). Above the pzc of silica, there is coulombic attraction between cationic surfactants and the negatively charged solid surface. At the same time, PEG can adsorb on the silica surface through hydrogen bonding (27-34), which transfers the particles into the top phase. Since dextran has only a poor affinity for SiO$_2$ (38-41), the polysaccharide adsorption is not so important. Thus, a competitive adsorption process occurs in this system when the particles are dispersed in the solution. Therefore, the solid partition is mainly determined by the relative strengths of the PEG/SiO$_2$ and DTAB/SiO$_2$...
interactions. Around $pH_{PZC}$, most of the surface silanol groups remain uncharged. The electrostatic attraction is not favored, and the hydrogen bonding may predominate in the system. Therefore, silica particles prefer the PEG-rich phase. As $pH$ increases, the surface becomes increasingly negatively charged, and then the electrostatic attraction between the cationic surfactant and the solid begins to dominate in the system. Thus, as $pH$ increases, the oxide particles begin to transfer from the top phase to the interface, and then to the bottom phase.

**Effect of pH on Silica Partition in the Dex/TX100 System**

Figure 3 shows the partitioning behavior of silica in the Dex/TX100/H$_2$O system. Below $pH$ 3.5, almost all the silica particles went into the top surfactant-rich phase. When the $pH$ increased from 4.0 to 8.0, the particles began to concentrate at the interface, and the yields in the top phase decreased with increase in $pH$. Between $pH$ 9.0 and 11.3, the solids preferred the bottom polymer-rich phase. At $pH$ 11.5 and 11.7, the oxide particles mainly stayed at the interface. With further increase in $pH$ from 11.8 to 12.5, the solids transferred to the top phase again. For the cases where the solids accumulated at the interface, this portion of particles was combined with the silica in the bottom polymer-rich phase for purposes of mass balance.

It is known that nonionic surfactants, such as Triton X-100, can adsorb on silica surface in aqueous solution (45-53). The oxyethylene groups of TX100 molecules attach onto silica surface through hydrogen bonding with the neutral silanol surface groups. The surfactant molecules form surface micelles at the solid/liquid interface through hydrophobic interactions among the tail groups of the surfactant. Previous discussion shows that dextran can also adsorb on silica with a poor affinity (38-41).

According to the results available in the literature, in aqueous solution both dextran (38-41) and Triton X-100 (45-53) can adsorb on silica through hydrogen bonding. Therefore, when silica particles are dispersed in the Dex/TX100 system, similar to the PEG/Dex system, a competitive adsorption process occurs at the solid/liquid interface between the polymer and surfactant molecules. Therefore, the partition behavior of silicon dioxide is determined by the relative strengths of the Dex/SiO$_2$ and TX100/SiO$_2$ interactions. Around $pH_{PZC}$ of 2 (43), most of silanol groups on the silica surface remain neutral. Both the polymer and the surfactant can interact with the solid through hydrogen bonding. From the above discussion, the TX100/SiO$_2$ interaction may be stronger than the Dex/SiO$_2$ interaction, because dextran only shows a poor affinity for the solid surface (38-41). Therefore, the particles prefer to stay in the top surfactant-rich phase. As $pH$ increases, the surface acquires negative charges through deprotonation of silanol groups, and the amount of neutral sites decreases. Since the oxygen atom in the oxyethylene unit of Triton X-100 is partially negatively charged, a certain level of electrostatic repulsion is introduced between the surfactant and the oxide. This repulsion force is demonstrated by the adsorption experiments in the literature which show that the uptake of TX100 by silica decreases with increase in $pH$ (45, 51). Thus, if follows that with increase in $pH$, the TX100/SiO$_2$ interaction becomes weak; it even may be less than that for Dex/SiO$_2$. The particles then begin to transfer from the surfactant-rich phase to the interface, then to the polymer-rich phase. This may explain the experimental trend in the $pH$ range of 3.5 to
11.3, i.e., the yield of the top phase kept decreasing with pH increase until most of the particles concentrated in the bottom phase.

Above pH 11.5, the solid partitioning behavior shows a strange trend, i.e., the particles preferred the top phase again. Similar to the results in the PEG/Dex system, this phenomenon is difficult to explain from current literature about adsorption of dextran and TX100 on silica. Therefore, it would be useful to undertake further study on the competitive adsorption of TX100 and dextran on silica surface at this pH range.

**Effects of Ionic Surfactants on Silica Partition in the Dex/ TX System**

*SDS.* Table 1 shows the effect of SDS concentration on silica partition in the dextran/Triton X-100 system at pH 3, 8, and 10. It can be seen that at low concentration (0.1 g/L), SDS had no effect on silica partition. At pH 3 and 8, 0.4 g/L SDS was enough to change the distribution of the solid. However, the particles always stayed in bottom phase, even up to 1.0 g/L. In Figure 4, the silica partition has been studied in a broad pH range from 2 to 12 with 0.4 g/L and 1 g/L SDS. With 0.4 g/L SDS, silica particles stayed in the top phase at pH 2; at the interface from pH 3, 5, and 12; and in the bottom phase at pH 7 and 10. At high SDS concentration (1 g/L), silica particles stayed in the bottom dextran-rich phase from pH 8 to 12. However, below pH 7, this amount of SDS induced the whole system to form only one phase.

In the polymer/nonionic surfactant aqueous biphase system, ionic surfactants mainly concentrate in the top surfactant-rich phase. They form mixed micelles with the nonionic surfactant. Therefore, a weak charged environment is introduced into this kind of aqueous biphase system (24-26). Addition of SDS makes the micelles in the top phase negatively charged. In the Dex/TX100 system, the partition of silica in the top phase at certain pH values is due to hydrogen bonding between the oxyethylene groups of the nonionic surfactant and silanol groups on the solid surface. Above pH\(_{\text{PZC}}\), it is expected that there is electrostatic repulsion between the negatively charged mixed micelles in the top phase and the similarly charged solid particles. Adsorption experiments show that TX100 adsorption on silica surface is suppressed from a mixed solution of TX100 and SDS (54). Therefore, the particles are expected to move from the top phase to the interface or bottom phase. The experimental results showed this really happened. As can be seen in Figure 4, at pH 12 (where silica stayed in the top phase in the absence of ionic surfactant) the oxide particles moved to the interface with 0.4 g/L of SDS, and even transferred into the bottom phase with 1 g/L SDS. From pH 3 to 5, the yields of solid in the top phase decreased in the presence of SDS. In the pH range of 9 to 11 (in which silica particles stayed in the bottom phase in the absence of SDS), the presence of SDS had no effects. At pH 2 (around the pzc), the solid surface is almost neutrally charged, and therefore, the coulombic interaction is not expected to be strong. Accordingly, the particles still preferred the surfactant-rich phase with addition of SDS.

*DTAB.* Table 2 and Figure 4 show the partition of silica in the dextran/Triton X-100 system in the presence of DTAB. From Table 2, it can be seen that 0.2 g/L DTAB was enough to transfer the silica from the bottom polymer-rich phase to the top phase. Figure
4 presents the silica partitioning behavior in the Dex/TX100 system from pH 4 to 11 in the presence of 0.4 g/L DTAB. In this pH range, silica always stayed in the top phase.

In the Dex/TX100 system, as with SDS, DTAB prefers the top surfactant-rich phase and forms mixed micelles with TX100 (24-26). Thus, a weakly positively charged environment is formed in the top phase. Electrostatic attraction is then expected to occur between the micelle in the top phase and silica surface. From mixed solutions of DTAB and TX100, adsorption experiments show a synergistic effect at low concentrations and an antagonistic effect at high concentrations (55). Therefore, silica particles are expected to prefer the top phase in the presence of DTAB. The experimental results demonstrate this expectation. From pH 4 to 11, silica particles stayed in the top surfactant-rich phase with 0.4 g/L DTAB, irrespective of whether they were at the interface or in the bottom phase before addition of cationic surfactant.

**CONCLUSIONS**

The partition of solid particles in aqueous biphasic systems depends on the physicochemical interaction between the solid surface and liquid phase, rather than the bulk properties of the solid particles. In the polymer/surfactant or polymer/polymer two-phase systems, the interaction between phase-component compound and solid surface plays an important role in determining the solid partitioning behavior. In both the PEG/dextran and dextran/Triton X-100 systems, silica particles stayed in the top PEG-rich phase at low pH. With increase in pH, the solids moved from the top phase to the interface, then to the bottom phase. At very high pH, the particles preferred the top phase again. These trends are attributable to variations in the polymer/solid and nonionic surfactant/solid interactions.

Addition of ionic surfactants into these two systems introduces a weakly charged environment, since ionic surfactants concentrate into one phase, either the top phase or the bottom phase. Therefore, coulombic forces also play a key role in the partition of silica particles because electrostatic attractive or repulsive forces are produced between the solid surface and the ionic-surfactant-concentrated phase. For the PEG/dextran system in the presence of SDS, SiO₂ preferred the bottom dextran-rich phase above its pHₚ𝑺𝑪. However, addition of DTAB moved the oxide particles from the top phase to the interface, and then to the bottom phase, with increase in pH. This phenomenon is attributable to the fact that SDS and DTAB concentrated respectively into the opposite phase of the PEG/dextran system. On the other hand, in the dextran/Triton X-100 system, the ionic surfactants concentrated in the top surfactant-rich phase and formed mixed micelles with TX100. Therefore, addition of an anionic surfactant, SDS, moves the silica particles from top phase to the interface or bottom phase. On the other hand, DTAB, a cationic surfactant, attracts the particles to the top phase in the pH range from 4 to 11, where in the DTAB-free system the solids either stay at the interface or in the bottom phase.
REFERENCES

Figure 1. Effect of pH on the partitioning behavior of silica in the PEG/dextran system. (PEG: 7.0 wt%, dextran: 16.8 wt%, silica: 2.0 g/L)

Figure 2. Effect of pH on the partitioning behavior of silica in the top phase of the PEG/dextran system in the presence of SDS and DTAB. (PEG: 7.0 wt%, dextran: 16.8 wt%, silica: 2.0 g/L, SDS and DTAB: 1.0 g/L)
Figure 3. Effect of pH on the partitioning behavior of silica in the Dextran/Triton X-100 system. (Dextran: 9 wt%, Triton X-100, 11 wt%, silica: 2 g/L)

Figure 4. Effect of pH on the partitioning behavior of silica in the top phase of the Dextran/Triton X-100 system in the presence of SDS and DTAB. (Dextran: 9 wt%, Triton X-100, 11 wt%, silica: 2 g/L; SDS & DTAB: as indicated in the figure)
Table 1. Effect of SDS concentration on silica partition in the dextran/Triton X-100 system. (Dextran: 9 wt%, Triton X-100, 11 wt%, silica: 2 g/L)

<table>
<thead>
<tr>
<th>SDS conc. (g/L)</th>
<th>pH 3</th>
<th>pH 8</th>
<th>pH 10</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Top phase</td>
<td>Mainly interface, some top phase</td>
<td>Bottom phase</td>
</tr>
<tr>
<td>0.1</td>
<td>Top phase</td>
<td>Mainly interface, some top phase</td>
<td>Bottom phase</td>
</tr>
<tr>
<td>0.2</td>
<td>-</td>
<td>Interface</td>
<td>-</td>
</tr>
<tr>
<td>0.3</td>
<td>-</td>
<td>Interface</td>
<td>-</td>
</tr>
<tr>
<td>0.4</td>
<td>Mainly interface</td>
<td>Mainly bottom phase, some interface</td>
<td>Bottom phase</td>
</tr>
<tr>
<td>1.0</td>
<td>-</td>
<td>Bottom phase</td>
<td>Bottom phase</td>
</tr>
</tbody>
</table>

Table 2. Effect of DTAB concentration on silica partition in the dextran/Triton X-100 system. (Dextran: 9 wt%, Triton X-100, 11 wt%, silica: 2 g/L)

<table>
<thead>
<tr>
<th>DTAB conc. (g/L)</th>
<th>Experimental results at pH 10</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>Bottom phase</td>
</tr>
<tr>
<td>0.1</td>
<td>Mainly interface, some top phase</td>
</tr>
<tr>
<td>0.2</td>
<td>Mainly top phase, some interface</td>
</tr>
<tr>
<td>0.4</td>
<td>Top phase</td>
</tr>
</tbody>
</table>