

# Laboratory- and mill-scale study of surfactant spray flotation deinking

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**ABSTRACT:** As the cost of quality waste paper continues to escalate in response to an increased global demand for this finite resource, loss of saleable fiber within flotation rejects becomes both environmentally and economically unacceptable. The ability of surfactant spray technology to reduce fiber loss without detriment to pulp brightness gains has been demonstrated during both laboratory- and pilot-scale flotation deinking investigations. This paper documents the successful transfer of this technology to a single flotation unit within the deinking line of a mill producing newsprint from 100% secondary fiber. Initial results suggest that the loss of fiber across the unit was reduced by more than 50% without obvious detriment to final pulp quality.

**Application:** Paper mills could significantly reduce fiber loss and save costs using surfactant spray technology in flotation deinking.

During the past few years, the use of recycled fiber has increased dramatically due to the shortage of fiber resources and restrictive environmental regulations on paper landfills. Since the successful application of flotation in the paper recycling industry from the minerals industry in 1980s, flotation deinking has become a necessary process for ink removal from wastepaper in many paper recycling mills. However, many problems remain unsolved such as high secondary fiber and water losses, fiber contamination that causes poor fiber bonding, paper-machine foaming problems through direct contact with surfactants in flotation, and low efficiency in removal of small ink particles, etc. Therefore, effective and innovative technologies based on the mechanistic understanding of flotation processes are greatly needed to solve or alleviate the above problems.

## FLOTATION DEINKING CHEMISTRY

A successful flotation process typically involves three fundamental processes: detachment of the ink particles from waste fibers, effective adhesion of the ink particles onto air bubble surfaces, and removal of froth and ink particles from flotation cells. The roles of surfactants in flotation deinking have been discussed in detail in the literature [1–3]. Although surfactants play important roles, they will also cause some adverse effects on ink removal, fiber quality, and water reuse. For example, both hydrophobicity and ink removal efficiency will decrease by

the adsorption of dispersant and frother [4,5]. The residual surfactant in recycled fibers is another problem that decreases fiber-fiber bonding, increases foam stability during the papermaking process, and adversely affects printing.

Although frother agent is necessary in flotation deinking to obtain a stable foam, there is an optimum foaming agent concentration in fiber suspension for ink removal as observed by Eppler et al. [4] and in our previous studies [6–8]. Ink removal efficiency increases with an increase in froth stability, so that there is an increase in frother concentration in conventional flotation systems. Unfortunately, the increase in frother concentration in the pulp suspension will increase the adsorption of frother onto ink particles, resulting in a reduction of the surface hydrophobicity of ink particles and ink removal [4].

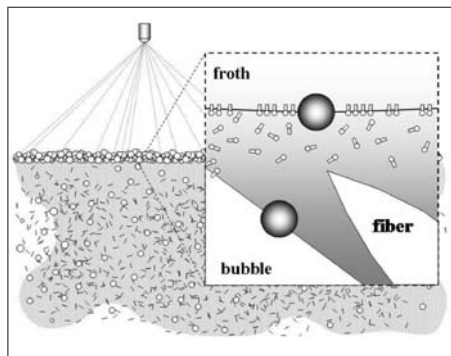
Previous researchers have studied the fiber loss mechanism of flotation systems. Some researchers postulated that fiber loss was due to fiber adhesion to air bubbles and then was removed with the froth [9–12]. Other researchers have challenged that postulation [13–16]. They found that the hydrophobicity of a fiber surface does not contribute to fiber loss. Instead, fiber loss is due to the mechanical entrapment of fibers in the froth.

In our recent study [6,7,17], we found that physical entrapment of fibers in an air bubble network and adhesion of hydrophobic parts of fiber surfaces on air bubble surfaces will contribute to the

total fiber loss. However, physical entrapment is the major contributor. It was also found that the fiber and water losses are directly related to the froth stability and froth structure. In general, a stable froth that often consists of small bubbles causes high fiber and water losses due mainly to the increased froth volumetric (or wet) rejection rate at an increased froth stability.

## SURFACTANT SPRAY FLOTATION

Surfactant spray technology represents a cost-effective means to impart some amount of process control to conventional flotation deinking operations. As the name implies, an aqueous frothing agent spray is applied onto the surface of an active flotation unit. The rationale of the foaming agent spray concept is that the foaming agent is used only to stabilize froth. By restricting the surface active frothing agent to the upper layer of the flotation unit rather than mixing it with the incoming furnish, this technology can avoid the dilution of the foaming agent by the bulk volume of the pulp suspension and the reduction in the hydrophobicity of the ink particles due to the adsorption of foaming agent onto the ink particle surface (Fig. 1). By simply adjusting spray composition and/or delivery rate, the technology may be customized to offset extremes in foam stabilities associated with sudden variations in the composition of the feed furnish. Furthermore, surfactant spray technology also makes it possible to separately control the foams



**1. By applying frothing agent as a spray to the top of a conventional flotation cell, unfavorable interactions within the bulk pulp suspension may be avoided.**

across a series of flotation cells by spraying different amounts of frothing agent to them. Therefore, it would be much more effective to apply it where foam needs to be stabilized.

Our previous studies demonstrated that fiber loss was reduced by up to 50% without affecting the ink removal efficiency when surfactant was sprayed on top of a flotation cell in deinking toner-printed papers in a bench scale column flotation cell [7] and in deinking offset old newspapers (ONP) and old magazines (OMG) in a laboratory-scale commercial flotation cell [8].

We believe the reduction of fiber loss was due to better control of foam stability, the foam structure and the spray washing effect that returned the fibers entrapped in the foam to the pulp suspension, as evidenced by Robertson et al. [18] in their experiments on foam washing during flotation.

Although the value of surfactant spray flotation deinking has been demonstrated at the laboratory and pilot-level [7,8,19], this paper documents the first attempt to extend the technology to full-scale operations.

## EXPERIMENTAL

### Materials

For this study, we used a Triton X-100, a nonionic octyl phenyl ethoxylate frother (Sigma-Aldrich, analytical grade,  $C_8Ph(EO)_{10}$ ; Ph = phenyl), and TDA-32, a polydimethylsiloxane-based defoamer emulsion (Taylor Chemicals, 65% solid), as received.



**2. The gas sparged, cyclone-type (GSC) flotation cell was fitted with a bank of 14 nozzles for delivery of the surfactant spray onto the surface of the aerated pulp.**

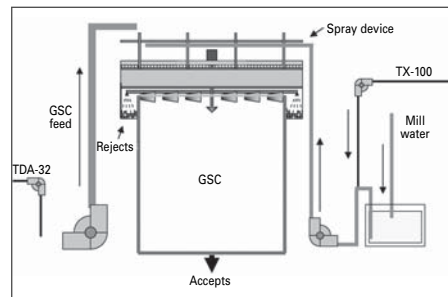
### Laboratory-scale trials

In preparation for the mill-scale trial, laboratory-scale flotation trials were conducted with the pulp obtained directly from the feed to the cell selected for application of the surfactant spray technology. The pulp included the deinking chemistry that had been added at the mill repulping process. To counteract the formation of the froth from the components of the whole furnish, we added defoamer into the pulp for use with the spray surfactant technology. This phase of the investigation was of particular relevance because the chemistry would also be present within the flotation unit feed during the actual mill trial.

**Flotation procedures:** The flotation trials were carried out using a Voith Sulzer flotation cell with a 18 L capacity (E-18) operating in circuit. The flotation process was conducted at the following conditions: 1% pulp consistency, 5 min flotation duration, 43°C, and 30 standard cubic feet per hour (SCFH) air flow rate. We collected rejects that overflowed from the top of the flotation cell during flotation for quantification of yield loss. We added enough defoamer to the slurry to counteract the frothing agent contained in the mill pulp slurry before the surfactant spray flotation.

### Surfactant spray device and conditions:

A surfactant spray system is located on the top of the flotation chamber of E-18 cell. Bottled compressed air drove the pressure swirl atomizers. The designed flow rate of the atomizers (Delavan, Inc., Des Moines, Iowa, USA) is 1.5 gallons per hour (gal/h). A TX-100 solution was used as the surfactant spray solution. Two atomizers were used in the E-18 cell. The atomizers, separated by 97 mm, formed a row 50 mm away from the aeration tube and perpendicular to the reject flow direction.



**3. The flow chart of surfactant spray mill trial.**

### Measurement of brightness, fiber loss:

We made 4.0 g filter pads using related Buchner funnel methods (PAPTAC Standard C.4U). Brightness was analyzed by TAPPI method T452 om-98. Yield loss was calculated using the ratio of the weight of the rejects (oven dry) to the weight of the waste paper (oven dry) at the beginning of flotation. For results analysis, we made five paper pads for brightness measurement and five paper pads for yield analysis.

### Mill trial

**Mill selection:** The mill selected for this study uses a 100% post-consumer furnish for newsprint production. Ink removal from the incoming feed is accomplished via two separate deinking lines incorporating conventional flotation equipment.

**Flotation unit selection:** Following an empirical analysis of the deinking operations and equipment used by the mill, a gas sparged cyclone-type (GSC; volume: 11,000 gallons) flotation cell (AhlFloat, Ahlstrom, Glen Falls, New York), was selected for the surfactant spray trial. The preference for this particular unit stemmed from the ease with which representative pulp samples may be taken from the feed, accept, and reject streams. In addition, background analytical data equated this unit with considerable fiber loss.

**Nozzle bank construction:** The nozzle bank consisted of a central manifold 4 in. PVC pipe from which 14 "L-shaped" 2 in. PVC pipe "arms" extended out over the cell surface (Fig. 2). This design placed the nozzles, mounted at the terminus of each arm, as close as possible to the cell surface without interfering with the regular rotation of the foam scraper. The lengths of the arms were varied to ensure that the nozzles, each capable of delivering 5.05 gallons per min (gal/min)

when operated at 20 psi (McMaster-Carr catalog number: 32885K221), would provide maximum, uniform coverage with minimum overlap of the full-cone spray patterns. The diameter of flotation cell is 19 feet and the average area covered by each nozzle is 1.88 m<sup>2</sup>. The pressure within the nozzle bank was maintained at 20 psi.

**Surfactant spray delivery:** In lieu of preparing a premixed surfactant solution before the trial (necessitating a container with a volume greater than 12,000 gal for a 3 h trial), we adapted a system to generate the solution *en route* to the nozzle bank. Fresh water, drawn from a 350 gal container by a centrifugal pump, was sent to the nozzle bank at a flow rate of 70.7 gal/min. The container volume was kept constant throughout the trial with fresh water. Undiluted Triton X-100 was metered into the suction side of the pump, across which adequate mixing of the miscible liquids was believed to occur. The dosing rate was adjusted to ensure that a 100 ppm TX-100 surfactant solution was pumped into the nozzle bank for delivery onto the aerated pulp.

**Defoamer addition:** Since the surfactant spray technology was incorporated within only one of the two deinking lines present at the mill, the deinking chemistry, introduced directly into the drum pulper (providing furnish to both lines) could not be suspended during the trial. To this end, the chemistry, complete with frothing agent, was present within the GSC feed. To counteract the foaming capacity of the whole furnish and ensure that any foam generated during the trial was a consequence of surfactant spray addition, the siloxane-based defoamer, TDA-32, was metered into the suction side of the GSC feed pump. To determine the minimal amount of defoamer needed to completely neutralize the foam, we established an expectedly high initial dosing rate of 200 ppm within the feed stream. This concentration was gradually reduced to the point at which foam could be neutralized with minimum defoamer consumption.

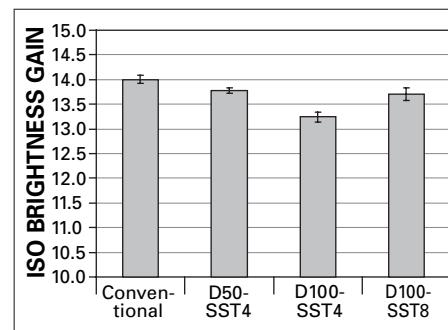
**Surfactant spray trial:** Figure 3 shows the flow chart of the surfactant spray trial. The trial commenced upon the establishment of dosing rates of

defoamer and TX-100 required for a stable, uniform froth on the cell surface. The foam flow rate and foam height should be controlled to be the same or a little lower than those of the conventional operation. Further detailed fundamental understanding of foam control can be obtained from our previous study [7, 8, 17]. Over the course of 3 h, samples were taken from the GSC feed, accept, and reject streams every 30 min. After determining consistencies of all three streams, we prepared brightness pads from the feed and accept streams according to TAPPI Standard Method T 218 sp-97. Brightness and effective residual ink concentration (ERIC) measurements were taken from three separate fields on both sides of five pads using a Technibrite TB-1C equipped with an ERIC 950 module (Technidyne Corp.). Total ash and carbonate contents were determined for samples taken from each stream according to TAPPI Standard Methods T 211 om-93 and T 413 om-93, respectively. For convenience, all oven-dry mass lost during combustion at 525°C was presumed to represent the cellulosic (i.e. fiber) fraction of the total solids. This allowed the consistencies of all streams to be reported as total solids and percent "fiber." We prepared five pads for the consistency measurement. Quantification of yield loss during the trial involved performing a simple consistency-dependent mass balance around the GSC at each time point.

## RESULTS AND DISCUSSION

### Laboratory-scale trials

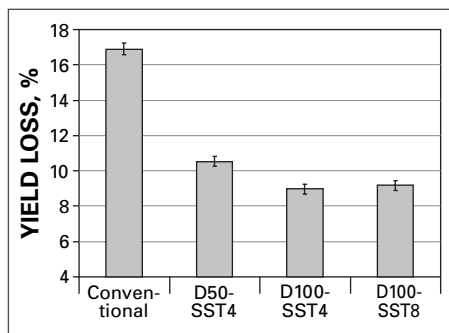
As noted, the rationale behind the foaming agent spray concept is to separately apply and control various deinking chemicals. However, the commercial deinking chemical currently used at the mill is a blend of different surfactants, which can act as both collector and frother. To reduce the fiber entrainment in the foam network, the role of foaming agent played by the commercial deinking chemicals must be suppressed. Required foams should be recreated and controlled by spraying a foaming agent to the top of the flotation cell. Therefore, we added defoamer into the pulp slurry to counteract the froth generated by the surfactant that was already added into the pulper at the mill. We used three



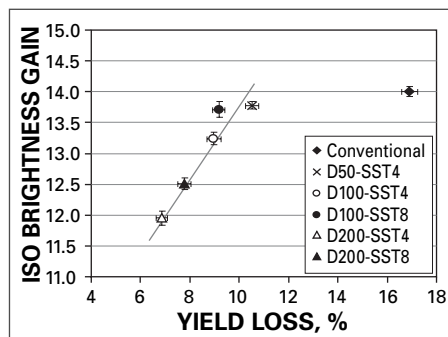
**4. Comparisons of brightness gains obtained from the pads prepared from the feeds and accepts during conventional flotation deinking and during the incorporation of surfactant spray technology.**

different loading levels of 50, 100, and 200 mg/L of TDA 32 defoamer (based on the total pulp slurry). Two concentrations, 100 and 200 mg/L, of TX-100 solution were used as the surfactant spray solutions. Total spraying amounts during flotation at those concentrations were 4 mg/L and 8 mg/L, respectively, on the basis of whole pulp furnish.

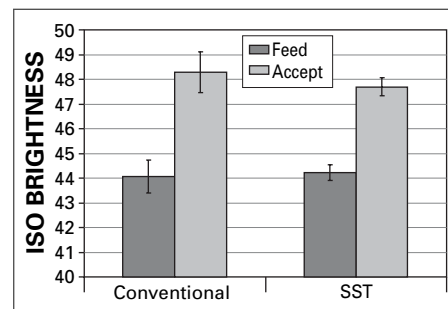
During the spray flotation process, froth generated by the deinking chemicals was suppressed by the addition of defoamer TDA-32. Foam volume and stability were reduced as the loading amounts of the defoamer increased. We also observed that the foam stability and structure very quickly responded to the amount of TX-100 frother sprayed, i.e. the amount of foam quickly increased as spraying amount increased and quickly decreased as spraying amount decreased. The response time was about a 10–20 seconds. Furthermore, ink removal efficiency and the final brightness of the recycled pulp were improved by the surfactant spray technology. Figure 4 shows the brightness gains obtained from the pads prepared from the feeds and accepts during the conventional flotation deinking with and without using surfactant spray technology at the laboratory-scale trials. The brightness gain of the pulp produced by the surfactant spray technology is about 0.3–0.5 ISO lower than that produced by the conventional flotation. However, significant reductions in fiber loss were also observed (Fig. 5).



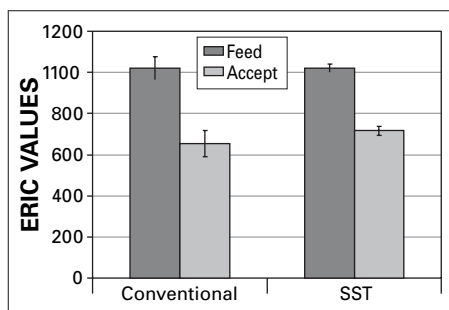
**5. Comparisons of yield losses accumulated across the flotation cell during conventional flotation deinking and during the trial incorporating surfactant spray technology.**



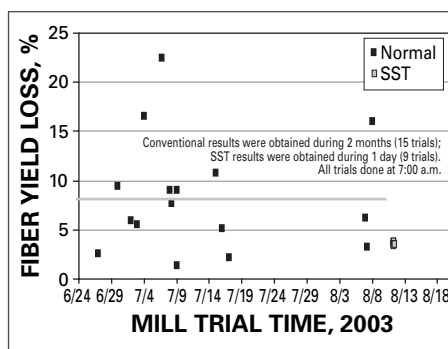
**6. Yield loss as a function of brightness gain values, presented for both conventional and surfactant spray technology.**



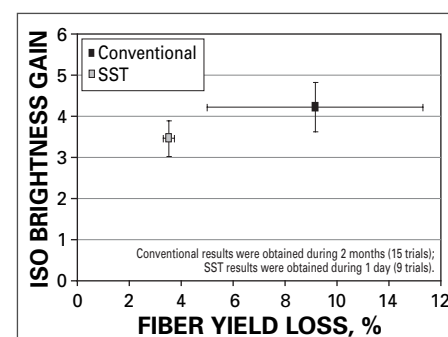
**7. Brightness values obtained from pads prepared from the GSC feed and accept streams during conventional flotation deinking and during the incorporation of surfactant spray technology.**



**8. ERIC values obtained from pads prepared from the GSC feed and accept streams during conventional flotation deinking and during the incorporation of surfactant spray technology.**



**9. Fiber yield losses obtained from pads prepared from the GSC reject streams during conventional flotation deinking and during the incorporation of surfactant spray technology.**



**10. Average fiber yield loss as a function of average brightness gain values is presented for both conventional and surfactant spray flotation deinking.**

Neither ink removal nor fiber loss can be used as a single measure to evaluate the performance of the deinking process because recycling mills need both high ink removal and low fiber loss. **Figure 6** shows brightness as a function of fiber loss obtained by both conventional and spray technology. Although the ~0.3–0.5 ISO brightness gain reduction was associated with the surfactant spray technique, this small deduction was considered tolerable in view of the remarkably reduced yield losses. D100-SST8 (100 ppm defoamer TDA-32 and 8 ppm spraying amount of TX-100 frother) provided the optimal results compared with the other conditions of this set of experiments as far as the ration of yield loss vs. brightness gain is concerned. Yield loss was reduced by 45.6% (from 16.9% to 9.2%) without obvious detriment to the pulp brightness gain (–0.3).

The foam generated at the surface of the pulp slurry is different from that generated inside the pulp slurry. That is, the fibers are more easily entrapped in the foam generated inside of the pulp slurry, but less likely to be entrapped by the foam stabilized at the surface. Therefore, the much lower fiber loss using surfactant spray technology compared with the conventional method at similar brightness gain is not surprising.

### Mill-scale trials

On the basis of flow volume and weight fraction, the amount of defoamer metered into the feed to effectively prevent foam formation was determined to be 7.3 ppm or ~1 lb per ton of oven-dry solids. When operated at 20 psi, the frother delivered onto the pulp amounted to 0.083 g/kg of oven-dry pulp (0.17 lb/ton).

**Figures 7 and 8** present the brightness and ERIC values obtained from pads prepared from each stream. The slight drop

in pulp brightness (~0.5) correlates with results observed during the laboratory-scale trials. This minor detriment may be readily managed within mills using a series of flotation cells or through optimization of the surfactant spray concentration, delivery rate or spray pattern.

**Figure 9** shows the yield losses obtained during the conventional flotation deinking and during surfactant spray deinking at mill-scale trials. The ability of the surfactant spray technology to dramatically reduce both values is immediately evident. In fact, the average yield loss during the trial was less than half of the average yield loss that occurred during conventional operations. Analysis of the fundamental furnish solids (i.e. fiber/fines and the ash fractions of both carbonate and non-carbonate nature) indicated that surfactant spray technology did not significantly alter the weight-fractional composition of total solids within the accept or reject streams

(Table I). Hence, this technology uniformly reduces the yield loss of these fundamental components of the furnish without affecting flotation removal rates of ink.

Yield loss, plotted as a function of brightness gain (Fig. 10), underscores the quantitative gains afforded by the surfactant spray approach to flotation deinking. Interestingly, the breadth of the results obtained during conventional operations suggests that brightness gain has limited correlation with yield loss. Conversely, the relatively low scatter obtained during surfactant spray trials confirms the technology as an effective means of process control.

## CONCLUSION

During laboratory- and mill-scale trials, surfactant spray deinking technology was found to improve yield across a single flotation unit. Although the brightness gains and ERIC reductions within the post-flotation pulps were slightly lower than those floated under conventional deinking conditions, the difference was viewed as tolerable in light of the remarkably reduced yield losses. In addition, the technology has demonstrated the ability to better control the flotation process, particularly the foam characteristics across a series of flotation cells. Both benefits, reduced yield loss and improved process control, were realized with minimal capital expenditure and equipment modification. It must be stressed that this initial mill-scale trial was conducted under conditions found optimal during laboratory—not mill—conditions. To this end, the system is expected to be far from optimized. Possible variations in future trials may involve modifying the nozzle-bank

|                  | CONVENTIONAL |        |         | SST  |        |         |
|------------------|--------------|--------|---------|------|--------|---------|
|                  | FEED         | ACCEPT | REJECTS | FEED | ACCEPT | REJECTS |
| Fibers/fines, %  | 86.2         | 87.5   | 45.4    | 86.5 | 87.8   | 40.7    |
| Carbonate, %     | 1.6          | 1.4    | 9.9     | 1.7  | 1.4    | 11.2    |
| Non-carbonate, % | 12.2         | 11.2   | 44.6    | 11.8 | 10.8   | 48.1    |

## I. Percentile composition of the total solids isolated from the feed, accept and reject streams during both conventional and surfactant spray (SST) flotation.

design (i.e. spray delivery rate, nozzle number, spray pattern, etc.) and the composition of the spray itself (e.g. replacing Triton X-100 with a more environmentally benign frothing agent). **TJ**

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## INSIGHTS FROM THE AUTHORS

For the current flotation process in paper recycling, many problems remain unsolved. These problems include high secondary fiber and water losses, fiber contamination that causes poor fiber bonding, paper-machine foaming problems through direct contact with surfactants in flotation, and low efficiency in removal of small ink particles. This paper introduces effective and innovative technology based on the mechanistic understanding of flotation processes to solve or alleviate these problems.

Although the ability of the surfactant spray flotation deinking has been demonstrated at the laboratory and pilot-level in our previous studies, this paper documented the first attempt to extend the technology to full-scale mill operations. The most difficult aspect of this research was the nozzle-bank design and installation; we successfully accomplished this design with the cooperation of the mill engineers.

The most important part of our findings was that, during laboratory- and mill-scale trials, surfactant spray

# RECYCLING

deinking technology was found to improve yield across a single flotation. Moreover, the technology has demonstrated the ability to better control the flotation process, particularly the foam characteristics across a series of flotation cells. Both benefits, reduced yield loss and improved process control, were realized with minimal capital expenditure and equipment modification. We feel that recycling paper mills could significantly reduce fiber loss and reduce costs using surfactant spray flotation technology to recycle ONP/OMG furnish.

It must be stressed that this initial mill-scale trial was conducted under conditions found optimal during laboratory conditions, not mill conditions. To this end, the system is expected to be far from optimized. Possible varia-

tions in future trials may involve modifying the nozzle-bank design (for instance, the spray delivery rate, nozzle number, or spray pattern) and/or the composition of the spray itself (for example, replacing Triton X-100 with a more environmentally benign frothing agent).

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**Zhao**



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**White**



**Zhu**



**Prein**