

Repurification of Process Condensate

In addition to a 35% savings in regeneration chemicals, repurification and reuse of process condensate substantially reduces water effluent.

Harvey D. Spangler
Farmland Industries, Inc.
Fort Dodge, Iowa

This article covers our experience with repurification of process condensate from a 600 ton/day ammonia plant and the reuse of it as boiler feed water to the unit. In our plant at Fort Dodge, Iowa, reuse of the process condensate increases the run lengths of the demineralizer and reduces the effluent water from the plant about 35%. This system has now been in operation approximately 16 months. Some of the equipment that was installed and originally believed to be necessary was later found unnecessary. As a result, a simplified recovery system was installed in our Dodge City, Kansas plant. The Dodge City plant has been repurifying their process condensate now for six months without any operating problems.

Process condensate is produced in a steam reforming ammonia plant when the unreacted steam in the process gas is condensed. Excess steam is required in the reformer in order to prevent the formation of carbon on the reforming catalyst. The excess steam which is not used in the reaction is condensed before the raw gas is sent to the carbon dioxide removal system. A 600 ton/day ammonia plant produces about 120 gal./min. of process condensate and a 1,000 ton/day plant about 200 gal./min.

The process condensate normally contains several impurities which are produced or have leached out of the materials in the reformer and other equipment. Ammonia is formed in the secondary reformer and the first stage shift converter. This ammonia is dissolved in the condensate which is also saturated with carbon dioxide. The process condensate also contains an appreciable quantity of methanol. This is formed in the second stage of shift conversion where coper is present in the catalyst.

In addition to the ammonia and the methanol, the process condensate also contains other organics and metals. These include amines which are produced from the reaction of ammonia and methanol and sodium, iron, copper, zinc, calcium, magnesium and silica, which are leached from the catalysts, refractory, vessel walls and piping.

Steam stripping can remove some impurities

The ammonia and organic impurities can be partially removed by steam stripping using a stripper tower and a reasonable quantity of stripping steam. Steam stripping reduces the quantity of the ammonia and carbon dioxide in the condensate so that complete removal by ion exchange is practical. Some of the methanol and other organics are also removed.

A typical analysis of the process condensate from the

raw gas separator shows that it normally contains about 900 ppm total nitrogen, principally as amines; and about 1,200 ppm methanol. The condensate stripper tower effluent normally contains about 20 ppm ammonia, 60 ppm methanol and about 40 ppm carbon dioxide.

Packed tower for stripping

Our process condensate stripping tower is a packed tower with two 15-ft. beds of Pall rings. The process condensate to the tower is preheated to about 195°F. by heat exchange with the tower bottoms. About 0.7 lb. of stripping steam is used per gallon of condensate. The tower is presently operating at near its maximum rate so that tower size limits the stripping steam rate and the impurities concentration in the bottom. A larger tower with more theoretical stages would enable more steam to be added and lower the level of these impurities further.

We felt that it was necessary that the ammonia be removed from the stripped process condensate by ion exchange prior to use as boiler feed water in the 1500 lb./sq.in. gauge steam system in our plant. Admiralty tubes are used in all of the surface condensers, and there was a possibility that these would be subject to attack by the ammonia.

The effect of the methanol was considered and there is no apparent problem with it in either the demineralizer or the steam system. Resin manufacturers confirmed that there should be no adsorption, fouling, or degradation of resin by methanol. In fact, methanol is frequently used as a solvent for removing organic foulants from resins.

All of the trace metals in the process condensate are removed by the demineralizer. These include the sodium, iron, copper, zinc, calcium, magnesium and silica. The cation resins in our demineralizer are normally regenerated with sulfuric acid. To insure that the heavy metals are removed from the resin, we have started to regenerate the resins once per year with hydrochloric acid to dissolve the metals.

Activated carbon removes heavy organics

The effect of the heavy organic compounds in the process condensate was considered and based on the data available at the time, we decided to remove them by adsorption using activated carbon. There was some concern that these organics may foul demineralizer resins and may result in foaming in the high pressure steam system. Also influencing our decision to remove them was the fact that we had two carbon drums in the unit which were no longer being used.

Data was obtained which showed that activated carbon would adsorb from 5 to 8% by weight of methanol. Hydrocarbons are selectively adsorbed by carbon with the heavier molecular weight compounds easiest to adsorb. Our carbon drums remove about 60% of the methanol and all of the heavier hydrocarbons when regenerated with steam on a monthly basis. The carbon drums have been bypassed several times to determine the effect of the organics on the 1500 lb./sq.in. gauge steam system. There does not appear to be any effect. The process condensate recovery facility at Dodge City does not have a carbon adsorption system since it does not appear to be necessary.

Our demineralizer utilizes a four-bed system with a strong-acid weak-base, strong-acid and strong-base vessels. The effluent from the strong-base then goes through a mixed bed for sodium removal. The stripped and cooled process condensate is mixed with filtered clarified water ahead of the primary cation exchanger to lower the temperature of the mixed stream to about 100°F. If the process condensate is to be demineralized without cooling, care must be taken in the selection of the weak base resin, since some of this resin has a maximum operating temperature of about 100°F. It is most advantageous to demineralize the stripped process condensate at as high a temperature as possible, since the demineralized water must be reheated for use as boiler feed water.

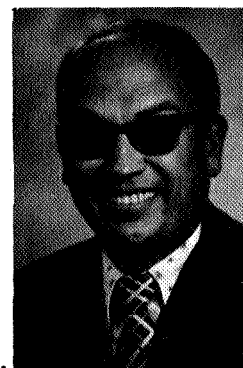
We have investigated the possibility of purifying the stripped process condensate in a separate system. This appears to be feasible using a weak acid cation resin followed by degasification for carbon dioxide removal. The demineralizer could be operated at about 175 degrees using a resin such as Rohm & Haas IRA-84. The effluent from the degasifier would probably need to be run through a mixed

bed to remove the metals and any carbon dioxide which leaks through the degasifier. Use of this system would increase the capacity of our demineralizer from about 320 gal./min. to about 450 gal./min.

In addition to about a 35% savings in regeneration chemicals in our plant, repurification and reuse of process condensate substantially reduces the water effluent from the plant. The effluent water from a 600 ton/day ammonia plant is normally about 600,000 gal./day. If the process condensate is repurified, the effluent water is reduced to about 400,000 gal./day. The amount of solids in the effluent water is not changed, so that the concentration of solids in the effluent water is higher when the process condensate is reused. The amount of ammonia in the effluent is not changed if ion exchange is used for purification, since the ammonia that is taken out by the resin is reintroduced into the effluent water when the resin is regenerated. #

Reference

1. Lombard, John, Catalyst & Chemicals Inc., Jan. 20, 1972. Private letter.



SPANGLER, H. D.

DISCUSSION

Q. Have you done any work on condensing the vapors off the stripper or do you just vent it to the atmosphere?

SPANGLER: We just vent the vapors to atmosphere. The vapors off the stripper that go to the atmosphere are only about one percent ammonia.

Q. Does most of the methanol go in the stripper overhead to the atmosphere?

SPANGLER: Yes.

GENE COMEAU, Farmland Industries: In your paper you make a statement about nitrogen being present as an amine.

SPANGLER: Yes.

COMEAU: Would you clarify that?

SPANGLER: The form of the nitrogen that is in the process condensate depends on several process conditions but it is normally between 75 and 90% amines in our unit.

COMEAU: Not ammonia.

SPANGLER: No, not ammonia.

COMEAU: How does it strip as amine or as ammonia?

SPANGLER: I couldn't tell you but it probably breaks down again into ammonia and methanol. I couldn't say that this breakdown occurs for sure but it is possible.

COMEAU: But it goes out the top of the stripper tower.

SPANGLER: Right.

COMEAU: Does the analysis for ammonia nitrogen show this.

SPANGLER: Right.

COMEAU: The analytical procedure for ammonia nitrogen will show this. Why do you think it's amines then?

SPANGLER: Process condensate samples were run from about 10 plants to determine what impurities were in the condensate. The quantity of amines that's in the condensate depends on a lot of things, like the activity of the low temperature shift catalyst and the steam to gas ratio. The formation of amines is apparently quite easy from the methanol and the ammonia that's present in the low temperature shift effluent.

Q. You said that you didn't want this ammonia in your 1500 pound boiler on account of Admiralty tubes. Suppose you didn't have any Admiralty tubes; how much ammonia would you put in your 1500 pound boiler?

SPANGLER: Well, I really couldn't answer that. I think Jim Finneran has done some work on that subject so you may wish to talk to him. I wouldn't be afraid of some ammonia in a 1500 pound steam system myself, but how much, I really couldn't say.