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Spent Palladium on Alumina Catalyst Regeneration for Hydrogen Peroxide Production

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Abstract: Catalysts used for the production of hydrogen peroxide deactivated more or less quickly with passage of time. Fixed bed reactor are designed for the production of hydrogen peroxide on large scale .There are many causes of catalyst deactivation, which may be the damage of active surface structure and dispersion, coke formation and the contamination by various chemicals which adsorb on the active sites. Coke is the term used for these large molecules often containing polyaromatic rings and which partially covers the active sites, as well as may block the catalyst porosity Spent palladium on alumina catalyst is calcined to maximum of 200-300 degree C to open blocked pores of catalyst.

Keywords: Regeneration, Calcination, Degradation, Palladium on alumina

Introductions

Production of hydrogen peroxide using fixed bed reactor by utilizing a palladium on alumina catalyst, which have palladium catalyst with contents 0.28 to 0.30 Wt %[1].The Palladium on alumina catalyst for hydrogen peroxide production must fulfill number of requirements such as:

a) Good abrasion resistance.
b) Working life should be long.
c) Easily regenerating.
d) Good productivity.
e) High selectivity.

After useful life of 2 years its activity decreased to about 30% fresh catalyst activity because of excessive reduction and oxidation [1][2].Deactivation of palladium on alumina catalyst is caused by deposition of high molecular weight organic materials on active sites of palladium on alumina catalyst formed from the polymerization of Ethyleanthraquinone [1][3][7]. The palladium on alumina catalyst also deactivated during hydrogenation process for the production of hydrogen peroxide by poisoning chemicals used for the production of hydrogen peroxide such as trimethylebenzene, trioctylephosphate and tetrabutyle urea, coke deposition and sometimes leaching of palladium on alumina catalyst[1][3][4].

Deactivated palladium on alumina catalyst regenerated with ethanol, nitric acid, boiling water, steam treatments and hydrogen peroxide [5].Active surface of palladium on alumina catalyst decreased in hydrogenation and regeneration cycle due to palladium migration outside to inside of pellets of catalyst [6][7] .To regain catalyst activity various treatments used such but most effective is calcinations of palladium catalyst .

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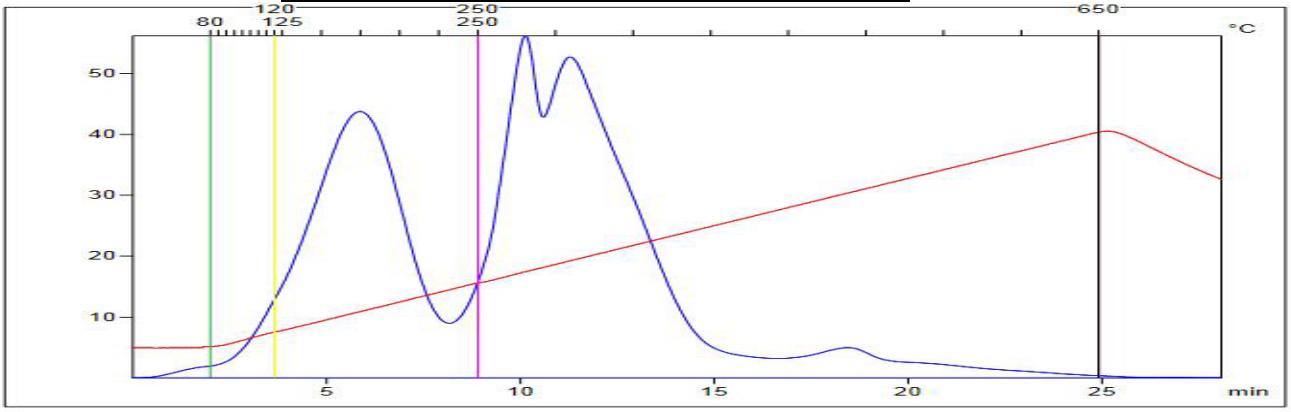
Experiments:

Spent palladium on alumina catalysts are regenerated by using a calciner system on a periodic basis .Active sites of palladium on alumina catalyst blocked with organic material removed in this way to regain its reactivity .Regenerated catalyst after passing through calciner screened and then washed with to remove any other origin material. As the spent catalyst contain high amount of free hydrocarbons, a stripping step was performed prior to regeneration. Stripping was efficient as no more free hydrocarbons are present on the stripped catalyst

Results and Discussions:

Main characteristic of used Palladium on alumina catalyst .Elemental composition is measured by XRF semi quantitative method. Potassium and phosphorous are in significant amount present on palladium on alumina catalyst. Shows in Table: 1.
During stripping, fraction of free hydrocarbons are transformed into carbonaceous material (coke), explaining the level of %C after stripping. This additional coke is easily removed during the regeneration step results are shown in Table: 2.
Due to high amount of free hydrocarbons on spent Pd catalyst, it has to be stripped prior to regeneration. After stripping + regeneration, catalyst has the main following characteristics (for details see the last section of the document).carbon and sulfur levels are below <0.1 wt% Palladium dispersion is 17%.

Table-1: Characteristics of Used Palladium Catalyst		
Carbon	wt%	22.4
free Hydrocarbons	wt%	20.6
Sulfur	wt%	0.1<
Elements present		
Pd	wt%	0.33
K	wt%	1.89
P	wt%	0.70
Na	wt%	0.08
Fe	wt%	0.03



Graph: Free hydrocarbons profile versus temperature on used palladium on alumina catalyst.

Blue curve: free hydrocarbons profile versus temperature (FID signal, arbitrary unit)

Red curve: temperature profile. Temperature could be read using scale on top of the figure.
Green, yellow and purples curves: used by lab technician to quantify free hydrocarbons by boiling po int zones.

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Table :2 Regenerated Spent Palladium on alumina Catalyst		
Carbon	wt%	0.1<
Sulfur	wt%	0.1<
Surface Area	m^2/g	95
Bulk crushing strength	Mpa	>1.6
Dynamic CO chemisorptions	mL CO /g	0.11
Corresponding Pd dispersion	%	17

Conclusion

Palladium on alumina catalyst used for the production of hydrogen peroxide is regenerated to restore its activity near or greater to fresh palladium on alumina catalyst. By increasing activity of palladium on alumina catalyst will economize hydrogen peroxide production process and life of the catalyst. Regenerated palladium on alumina catalyst has acceptable mechanical resistance.

Catalyst deactivation in hydrogenation caused several factors but causes are deposition of organic material which can be regenerated.

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