

# Investigation of Cement Slurry with Alkali Treated Sisal Fibers for Lost Circulation

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With the aim to challenge low plugging efficiency of plugging cement slurry caused by poor-dispersed plugging fibers in the cement slurry, the authors used alkali treated sisal fibers as plugging material of cement slurry and studied the plugging efficiency added a different dosage of alkali treated sisal fibers through the modified experimental set-up for lost circulation test. The microstructure, the types and quantities of polar groups in the surface of alkali treated sisal fibers were respectively evaluated by SEM and XPS. The results showed that the cement slurry added 0.3 % sodium hydroxide treated sisal fibers is well proportioned disperse and succeed in blockling less than 4 mm the size of pore or 4 mm the size of fracture. Mean while, the paper noted that the polar groups in the surface of sisal fiber are favourable for sisal fiber bridging and forming blocking zone in a simulated fracture or hole rapidly.

Key Words: Lost circulation, Sisal fibers, Alkali treated, Cement slurry, Dispersity, Plugging efficiency.

# INTRODUCTION

Well cementation is the process of placing cement slurry to the precalculated location in the annular space between casing and well bore with general functions to bond the casing and formation, to protect paying zone, to prevent the migration of formation fluids (oil, gas, water) between zones and to control lost circulation. The lost circulation is a concerned problem all over the world<sup>1-3</sup>. Many previous works have used materials such as mica, cellophane, walnut hulls, foamed cement and high strength micro sphere (HSMS) for lost circulation control<sup>4-6</sup>. But this type of material may result in permanent damage of paying zone. In recent years, during the construction of oil and gas well, fibers (nylon, polypropylene and other organic fibers) have been used in the cement slurry, not so much for reinforcement, but as a lost circulation material (LCM) to cure losses. However, the available organic fibers are difficult to properly disperse to cause floating or clumping on the slurry surface. The target of this study is to develop the sisal fibers (SF) cement slurry that can cure lost circulation and have no damage to the paying zone.

# **EXPERIMENTAL**

Sisal fibers with higher rigidity and the diameter of approximate 50-200  $\mu$ m were obtained from Guangxi Sisal Company, China. Sisal fibers was immersed in a solution of 10 % sodium hydroxide solution for 2 h at room temperature,

followed by water washing, air drying at 90 °C and cutting in 5-10 mm of length.

Raw materials were dispersing agent (USZ), fluid loss agent (G33), both from Weihui Chemical Engineering Co. Ltd., (Henan Province, China) and a API class G Portland cement (JHG) from Jiahua Group Co. Ltd., (Sichuan Province, China). Formulations of studied cementing slurry were 100 % Portland cement (JHG), 56 % dispersing agent (USZ), 35 % fluid loss agent (G33). The studied cementing slurry was fabricated with a 0.44 water to cement ratio. The alkali treated sisal fibers (ATSF) were added to the studied cementing slurry in concentrations of 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 % by weight of cement (BWOC).

All tests of cementing slurry were performed in accordance with ISO 10426-1 (2005) and simulated reservoir conditions in order to determine the effect of amount and length of sisal fibers on suitable cement system performance for lost circulation control during fractured formation cementation<sup>7</sup>.

A modified high-pressure, high-temperature (HPHT) API fluid-loss cell was used to test sisal fibers as lost circulation materials in cement slurries. At the top of steel ball a perforated or slotted plate was fitted. The sisal fibers cement slurry is poured from the top of cell to fill the cell column. This set-up is to check the plugging efficiency of sisal fibers in cement slurry from passing through either a perforated or slotted plate. Two types of plates were used. The perforated plate with holes of 2, 3 and 4 mm in diameter, respectively, simulated the

different pore sizes of the formation. The slotted plate had a rectangular slot (Fig.1) cut in it. The slot size was 50 mm long and 1, 2, 3 and 4 mm wide, respectively, to simulate different fracture sizes.



Fig. 1. Experimental set-up for lost circulation test

The laboratory test procedure was as follows. In each case, 400 mL of cement slurry added a given volume of sisal fibers were prepared and poured into the test cell (Fig. 1). The cell was closed and pressure of 7 MPa was applied. This operating mode was done to simulate the differential pressure created in the well when the fluid enters a fracture. The sisal fiber cement slurry was left under constant pressure for 0.5 h and the filtrate that passed through the plate was collected. The volume of filtrate collected provided an indirect measurement of plugging efficiency of the different cement slurries and the effect of the concentration of fibers. All the tests were carried out at 90 °C.

# **RESULTS AND DISCUSSION**

**Morphology of sisal fibers:** Physically, each fiber cell comprises cellulose, hemicelluloses, lignin, pectin and a small amount of waxes and fat.<sup>8,9</sup> This surface alkali treatment process is the common method to increases the number of possible reactive sites and allows better fiber wetting.<sup>10-14</sup>

Fig. 2 shows SEM (JSM-6490, Jeol) of the microstructure of sisal fiber. As can be seen, the surface of untreated sisal fiber was closely wrapped with a great amount of impurities which are lignin and hemicelluloses of sisal fiber cell. However, due to lignin and hemicelluloses removed during the alkali treatment process, the degree of polymerization and the molecular orientation of the surface of alkali treated sisal fibers are improved by alkali reagents. It was demonstrated that alkali treatment changed the morphology of the fibers by removing cellulosic and colloid, creating voids and producing fibrillation of fibers. These effects lead to a better adhesion between fibers and matrix.<sup>15</sup>



Fig. 2. SEM images of the sisal fiber (a) untreated; (b) alkali treated

**XPS analysis:** X-ray photoelectron spectra were collected using a Leybold MAX-200 X-ray photoelectron spectrometer (Leybold, Cologne, Germany). A high-resolution scan was conducted on the C1s region for untreated sisal fibers and alkali treated sisal fibers to determine the types and amounts of carbon-oxygen bonds present (Fig.3). The C1s peak was deconvoluted into three sub peaks, C1 (Binding energy 285.0 eV), C2 (Binding energy 286.5 eV) and C3 (Binding energy 288.0 eV).

Differences in the oxidized-to-unoxidized carbon ratio for untreated sisal fibers and alkali treated sisal fibers were apparent (Table-1). As expected, alkali treated modification significantly increased the concentration of carbon-oxygen bonds (C2 + C3) in the treated sample, proving that substantial surface oxidation occurred after 2 h of exposure to the 10 % sodium hydroxide solution. This was expected since alkali treated sisal fibers have a larger percentage of oxidized carbon in their chemical structure than sisal fibers. Due to increase of carbonoxygen bonds of alkali treated sisal fibers, the wettability of alkali treated sisal fibers was improved so as to homogeneous distribution of alkali treated sisal fibers in cement slurry.



Fig. 3. XPS of C1s region for the sisal fiber, (a) untreated, (b) alkali treated

TABLE-1 RELATIVE AMOUNT OF DIFFERENT CARBON-TO-OXYGEN BONDS AT SAMPLE SURFACE DETERMINED BY HIGH-RESOLUTION XPS								
Sisal fibers	Element	Atom content	Area	O/C ratio				
Untreated	C1s	86.76 %	2638.5	0.15				
	O1s	13.24 %	746.3					
Alkali treated	C1s	83.23 %	2318.4	0.20				
	O1s	16.77 %	865.8					

**Performance of cement slurry with alkali treated sisal fibers:** As can be seen from Table-2, generally, cement slurry in concentrations of 0, 0.1, 0.2, 0.3, 0.4, 0.5 and 0.6 % alkali

treated sisal fibers by weight of cement (BWOC) has less free fluid and fluid loss. A suitable cement system formulation for the lost circulation test is 0.1-0.5 % alkali treated sisal fibers (BWOC) with better compressive strength and flexural strength. The resulted data should be well presented by the form of schemes, figures, graphs, tables, reactions and equations. These items should be numbered clearly.

**Plugging efficiency of fiber cement slurry:** No changes in plugging efficiency observed above the fiber concentration of 0.3 % (by weight of cement) and hence 0.3 % is considered as optimum fiber concentration to control loss circulation problem. As can be seen from Fig. 4, leakage of all the cement slurry shows that neat cement slurry can't plug up 2 mm the size of pore or  $1 \times 50$  mm the size of fracture. By adding 0.3 % alkali treated sisal fibers the cement slurry can get comprehensive plugging effect and block less than 4 mm the size of pore or 4 mm the size of fracture.



Fig. 4. Plugging efficiency of cement slurry with alkali treated sisal fibers

Fig. 5 is the plugging effect diagrams of a 3 mm perforated plate and 3 mm slotted plate, the cement cake formed on perforated plate and slotted plate has been carefully removed with water leaving only the stoppage formed by the fiber cement in the hole or slot.

According to part imitation Fig. 6, it seems logical to surmise that alkali treated sisal fibers can easily distributed in homogeneity in the cement slurry. Compared to the previous plugging mechanism of organic fiber the surface of alkali treated sisal fibers has a large number of polar groups, which will form hydrogen bonded with vast hydroxyl in the cement slurry, thus making alkali treated sisal fibers generate network bridge over the fractures and creating the required filter cake in the leakage-channel due to friction, hang up and resort

TABLE –2 PERFORMANCE OF SISAL FIBER CEMENT SLURRY								
Dosage of ATSF (%)	Density (g cm <sup>3</sup> )	Free fluid (mL)	API fluid loss (mL)	90 °C × 24 h				
				Compressive strength (MPa)	Flexural strength (MPa)			
0.0	1.9	0	96.0	27.46	5.65			
0.1	1.9	0	136.0	24.37	6.225			
0.2	1.9	0	135.0	27.39	6.975			
0.3	1.9	0	96.0	24.28	6.30			
0.4	1.9	0	110.4	23.93	6.05			
0.5	1.9	0	91.2	23.23	5.975			
0.6	1.9	0	80.8	20.44	5.71			

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action of alkali treated sisal fibers on contact boundary of the cracks and pores more easily. The network structure can withstand pressure of 7 MPa, the cement particles will be silted in the irregular space of the fiber network structure and formed the compact plugging layers to bring about non-damaging to the formations and effective sealing to the leakage zone.



Fig. 5. Cement cake obtained after a lost circulation test through the 3mm hole plate (a) and 3\*50 mm slot plate (b) with a fibered cement slurry



Fig. 6. Simplified representation of alkali treated sisal fibers filled the fractures and formed a bridged to stop the losses

# Conclusion

The surface of alkali treated sisal fibers is under controlled condition not only removed a lot of pectin and hemicellulose, but also formed a large number of polar groups, which is propitious to increase wettability of alkali treated sisal fibers so as to homogeneous distribution in cement slurry. With the optimum dosage of 0.3 % alkali treated sisal fibers (BWOC), through the modified experimental set-up for lost circulation it proved that alkali treated sisal fibers with polar groups and higher rigidity is conducive to non-damaging to the formations and the improvement of plugging efficiency.

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